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THE ORGAN OF THE

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A MONTHLY JOURNAL DEVOTED TO THE ADVANCEMENT OF
ANALYTICAL CHEMISTRY.

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VOLUME XXVI.
1901.

BAILLIÈRE, TINDALL AND COX,
8, HENRIETTA STREET, COVENT GARDEN, LONDON, W.C., ENGLAND.

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ERRATA.

Page 229, 5th line from bottom, *for* "As the figures . . . ." *read* "As the figures given for a commercial malt extract must necessarily be of a purely empirical nature, it was decided . . . ."
Page 230, foot-note, *for* "author's" *read* "authors'."

ADDENDUM.

Page 264. The results in Mr. Richmond's paper are expressed in parts per 100,000.

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THE ANALYST.

JANUARY, 1901.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE monthly meeting of the Society was held on Wednesday evening, December 5, in the Chemical Society's Rooms, Burlington House. The President (Mr. W. W. Fisher, M.A.), occupied the chair.

The minutes of the previous meeting were read and confirmed.

Certificates of proposal for election to membership in favour of Messrs. Percy Henry Carpenter and Samuel Russell Trotman, M.A., were read for the second time; and certificates in favour of Messrs. A. Leonard H. Garside, Torbay College, Torquay, Analytical Chemist, Lecturer on Chemistry at the Vivian Institute, Torquay; Robert George Grimwood, F.I.C., 17, Dagmar Road, Stroud Green, London, N., chief assistant to Mr. W. J. Dibdin, and Gas Examiner to the London County Council; John Charles Umney, 48, Southwark Street, London, S.E.; and H. Rowley, 277, St. George's Terrace, Perth, West Australia, Public Analyst for Perth and Fremantle, were read for the first time.

Mr. John Stewart Remington was elected a member of the Society.

Dr. Lewkowitsch and Mr. L. Kidgell Boseley were appointed to act as auditors of the Society's accounts for the year 1900.

The following papers were read: "The Examination of Extract of Malt," by Walter J. Sykes, M.D., and C. A. Mitchell, B.A.; "Note on the Estimation of Glycerine," and "The Examination of Varnish Resins," by J. Lewkowitsch, Ph.D.; "Note on the Occurrence of Barium in the Spring Water of Boston Spa," by Percy A. E. Richards; and "On the Analysis of Samarskite," by Arthur G. Levy.

A discussion, opened by Mr. Alfred C. Chapman, took place on the methods of testing for the presence of arsenic in beer and brewing materials.*

Some condensers of special patterns were shown by Mr. C. T. Tyrer.

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* Mr. Chapman has, since the meeting, elaborated his remarks; they will be found on p. 8 of this number.

THE DETERMINATION OF THE AVAILABLE EXTRACT OF MALT.

By Lawrence Briant.

(*Read at the Meeting, November* 7, 1900.)

The determination of the available extract of malt is one in which there is room for considerable divergence of result. The operation is a very different one to the estimation of an acid or a base, for it is an endeavour in the laboratory to ascertain the amount of extract which is obtainable by a brewer in his actual brewing operations, the amount being dependent upon the specific gravity and quantity of saccharine wort obtainable from a known quantity of malt during the mashing and sparging operation in the brewery. My object in bringing this matter before you to-night is to point out the discrepancies which at present exist between determinations by different methods, and to suggest a method which is at once easy of performance and gives accurate results. Under the present condition of things, it is not an uncommon thing to find a difference of 2, 3, or even 5, pounds per quarter in the results given in different analyses. Such a difference would be a very serious matter to the brewer. Taking, for instance, a variation of 5 pounds per quarter (and this I have actually known to occur), and assuming that the brewer mashed 50 quarters of the malt per day, the difference in yield of beer per week of five days' working, represented by the divergence in the analysts' results, is no less than 62½ barrels of a 20 lb. beer. It is natural that brewers may think these discrepancies are proof of very careless work, yet the fact is that they are really due to the varying methods adopted in laboratories.

In attempting to estimate the extract yielded by a malt, the obvious method seems to be to imitate as closely as practicable the actual operations as carried out in a brewery. The brewer mixes his ground malt with water in about the proportion of 2 barrels of water to 1 quarter of malt—that is, 720 parts of water to 336 parts of malt. He allows this mixture to stand at a temperature depending upon the character of the malt and the class of beer which he is producing, but which lies between 145 and 155. At the expiration of a given time—generally two hours—saccharification being complete—he draws off the extract and washes out that which remains in the "goods." But this "sparging" operation does not merely consist of washing. The brewer sparges with water at a temperature considerably more elevated than that of his mash. As a consequence, the heat of the mash slowly rises, and starch, which during the malting operation has been imperfectly modified, gradually comes into solution, and the amount so dissolved will depend upon the original character of the malt, upon the temperature of the sparging water and the amount of the sparge. We have therefore a varying set of conditions which it is not easy to precisely imitate in the laboratory, and the extract yielded by a malt will depend a great deal upon these factors. The following results show this to be the case as regards temperature of stand of mash:

Mixture-heat.	Extract per 336 pounds.
140° F.	90·3 pounds.
145° F.	91·2 ,,
150° F.	90·1 ,,
155° F.	88·9 ,,
145° F. (raised to 150° by underlet in 20 minutes)	92·3 ,,

The extract will also, as above mentioned, vary with the temperature of sparging water. This is shown by the following figures:

Mixture-heat.	Sparge-heat.	Extract per 336 pounds.
149° F.	160° F.	91·3 pounds.
149° F.	175° F.	93·4 ,,
149° F.	⅓rd 185° F. ⅓rd 175° F. ⅓rd 165° F.	95·7 ,,

So that we see, by different temperatures of sparge, the extract may be affected to the extent of 4½ pounds per quarter.

Yet another condition influences the result, namely, the fineness of grinding. There is, unfortunately, no standard of grinding adopted by chemists, yet the degree of fineness much influences the result. This is seen in the following figures:

	Extract per 336 pounds.
Very coarse grinding	92·0 pounds.
Coarse ,,	94·4 ,,
Fine ,,	95·7 ,,
Crushed in mortar	94·7 ,,

It is quite evident that, as precise temperature of mixture heat—sparge heat and grinding all exert a considerable influence upon the extract—it is very difficult to get regularity of working upon these lines.

For many years the author was in the habit of determining the extract of malt in a miniature mash conducted on precisely the lines obtaining in breweries, though the influence of bulk was of course absent; but it was found that very slight alterations in temperature, in dilution of mash, in heat, and in amount of sparging water, considerably affected the results, so that it was necessary to take the average of several determinations; it was not always possible to know at what temperature the malt would be mashed or sparged. Owing to these difficulties, he has now discarded this method as more troublesome and less reliable than that which will be presently described.

A method which is very largely adopted is that of Heron, as described in a paper read before the Society of Chemical Industry (*Society of Chemical Industry*, 1888, vol. vii., p. 259), and afterwards modified in a paper read before the Institutes of Brewing, in 1895 (*Journal of the Federated Institutes of Brewing*, vol. i., p. 116). This method consists in weighing out 50 grammes of ground malt, transferring to a flask, and adding 400 c.c. of water at such a temperature that the initial mixture heat may be 122° F. The whole is immersed in a water-bath and the temperature gradually raised, so that at the end of an hour it has reached 158°. The mash is now raised to 190° for fifteen minutes, then cooled to 60°, and the whole made up to the bulk of 515 c.c. After being shaken, the liquid is filtered off, its specific gravity determined, and the extract calculated in the ordinary way, the assumption being made that the liquid occupies a bulk of precisely 500 c.c. There are several advantages in this method. The process is simple and rapid, no sparging is required; thus, errors due to that source are eliminated, whilst differences in mixture heat have very little influence upon the extract yielded, as is shown by the following figures:

Heron's Method.

Mixture-heat.	Extract per 336 pounds.
140° F.	95·2
150° F.	95
160° F.	95

It is a remarkable fact that whilst with thick mashes temperature exerts a very important influence on yield, with thin mashes practically no difference occurs.

But this method is not, in my opinion, entirely satisfactory. Heron, it will be remembered, makes up to a constant bulk of 515 c.c., yet there is a very great variation in the amount of husk contained in malt samples. This is abundantly evident to anyone who is accustomed to the analysis of malts, and the amount of husk present in a thin barley foreign malt is manifestly very much larger than that in a thin-skinned, plump, bold, English or Scotch barley malt. There is thus an error introduced due to this difference for which Heron has not made allowance. Further than this, the raising of the mash to 190° for fifteen minutes is open to the objection that, in practice, the mash never rises to such a temperature, very seldom (even during sparging) exceeding 160°.

Stern (*Journal of the Federated Institutes of Brewing*, vol. i., p. 448) proposes to overcome this source of error by making two mashes, both of which are mashed under Heron's conditions, but to one a known bulk of water is afterwards added. The specific gravity of the two filtrates is ascertained, and from that, by means of algebraic formula, it is possible to calculate the true extract. To those of us, however, who have in the course of a season to analyse a very large number of samples of malt, Stern's method is manifestly too laborious for practical application. The method which I propose eliminates the error arising from difference in amount of husk, and gives the true extract value of the malt sample. I proceed as follows:

Weigh out roughly a little over 50 grammes of the malt, and grind. Place the whole of the ground malt in a tared scoop, and accurately weigh 50 grammes. Now place the ground malt in an ungraduated ordinary boiling-flask of about 550 c.c. capacity, and run in as rapidly as possible a bulk of water at a temperature of 160° F., which shall be equal in volume to 400 c.c. if measured at 60° F. The measurement of this bulk of water is made in a flask or other suitable apparatus, graduated at the point occupied at 160° by 400 c.c. of water at 60°. Immediately thoroughly mix the contents of the flask. Loosely cork the flask to prevent evaporation, place in a water-bath maintained at a temperature of 150°, and allow to stand for two hours.*

---

* One hour's stand is usually sufficient, but for some malts a longer stand is necessary, and two hours' digestion insures complete extraction of all classes of malt.

At the end of this time cool the mash to 60° F., add 100 c.c. of water at 60°, mix thoroughly, filter bright, and take the specific gravity of the wort. We are now dealing with an actual bulk of 500 c.c. of liquid plus the volume occupied by the saccharine matter dissolved from the malt. It is necessary for us to ascertain what this volume is, and this we can calculate from the specific gravity of the liquid. To take an example: Suppose its specific gravity to have been 1028, now divide the excess weight over 1000 by the solution factor 3·86, = 7·25, the solid matter present in 100 c.c. of the wort. This multiplied by 5 (7·25 × 5 = 36·25) gives us the amount of solid matter (36·25) dissolved from the malt. Now, as 15·9 parts of saccharine matter occupy a bulk when dissolved of 10 parts, it will be seen that the bulk occupied by the saccharine matter dissolved from the 50 grammes of malt is 15·9 : 36·25 : : 10 = 23 c.c.

Our total dilution of mash is therefore 523 c.c., and this corresponds with the rate of 9·75 barrels per quarter. In order to further simplify the calculation, we may multiply our excess weight over 1000 of specific gravity direct by the factor 3·51, which gives the true extract per quarter of 336 pounds. Thus, 28 × 3·51 = 98·2 pounds per quarter of 336 pounds. In practice this factor may always be used. The variations in specific gravity which occur are not sufficient to materially affect the result.

The modified process, therefore, which I suggest, consists in mashing 50 grammes of malt with 400 c.c. of water at a mixture-heat of 150° F., standing for two hours, cooling to 60°, adding 100 c.c. of water, mixing, filtering, taking the specific gravity, and multiplying excess weight over 1000 by the factor 3·51.

Discussion.

Dr. Sykes said he perfectly agreed with Mr. Briant as to the necessity that some definite scheme for analysing malt should be agreed on. The process Mr. Briant had suggested seemed to him to be a very ingenious one, and likely to surmount many difficulties.

Mr. Arthur R. Ling said he thought most of those who had to make determinations of extract in brewery malts had found the method proposed by Mr. Briant in his well-known text-book too lengthy, although it undoubtedly gave results more in harmony with those obtained in actual practice than did the shorter methods. He had been in the habit of using the method devised by Mr. Heron, but, like Mr. Briant, he had had doubts whether the volume occupied by the grains from 50 grammes of the malt was always exactly 15 c.c., as it had been assumed to be by Mr. Heron. Recently, therefore, he had carried out the determination in a manner which did not necessitate such an assumption. Fifty grammes of the ground malt were added to 400 grammes of water, at the requisite temperature, contained in a tared beaker or flask. The mashing was conducted as usual, but, instead of ultimately making up the cooled mash to 515 c.c. with water, the beaker or flask was placed on a balance, and sufficient water weighed in that the total quantity (including that already present) amounted to exactly 500 grammes; the specific gravity of the filtrate was then determined as usual. It was only right to add that, as far as his experience went, the results agreed with those obtained when working in accordance with Mr. Heron's directions.

Mr. Julian L. Baker said that, as laboratory results were never absolutely representative of what took place in the mash-tun, it would be highly desirable that all brewing chemists should use one method for the determination of extract; and he personally preferred Heron's widely-used method on account of the very important desideratum—rapidity.

Mr. Chapman said that the failure to make a determination of the available extract of a given sample of malt, or the incorrect determination of such extract, might result, as Mr. Briant had shown, in a considerable loss to the brewer, owing to the fact that unconsciously he might be obtaining from his malt a much smaller proportion of soluble matter than it ought to yield. If the malt contained soluble substances only, which had simply to be extracted, the matter would be a very simple one; but the determination had to be based on the amount of soluble matter formed as the result of a series of chemical changes, influenced by a number of conditions, and an approximation ultimately arrived at which should as nearly as possible represent the truth. From his own experience, he was of opinion that there could be no such thing as "the brewery extract" of a sample of malt; and that fact—at any rate, it appeared to him to be a fact—had led him to adopt a process yielding numbers which were always in excess of those obtained in the brewery, but which bore to the brewery extract a definite and (by him) understood relationship. Given one and the same malt, two brewers—situated, it might be, in the same street—would obtain two different quantities of extract, both perhaps working correctly, but using different procedure. The extract depended upon the precise character of the starch contained in the malt, or, in other words, the amount of modification which the contents of the grain had undergone, and also upon the procedure in the brewery itself—whether the mash was allowed to stand for a longer or shorter time; whether the mashing temperature was higher or lower; whether or not an underlet was used; whether a strong beer or a weak beer was being brewed, all of which were factors in actual working procedure, which exercised considerable influence upon the extract obtained. The possibility of ever arriving at a correct laboratory estimation of the "brewery extract," except by a compensation of errors or something of that kind, seemed to be precluded by several considerations. For instance, uniformity of grinding, which was referred to by Mr. Briant, was quite impracticable in the case of a heterogeneous material like malt. Moreover, laboratory experiments were necessarily made on a small scale, whereas in the brewery the extracts were determined when all the operations were concluded. The specific gravity was not by any means strictly proportional to the concentration; and a distinct error was introduced in the calculation of the small laboratory numbers to the large numbers of the brewery. Perhaps, therefore, after all, a purely arbitrary method was as likely to give satisfactory results as one in which an endeavour was made to closely imitate actual brewery practice. In addition to the intrinsic interest of Mr. Briant's paper, it was, he felt, of value as a plea for uniformity in various branches of analytical work; and there was no department in which the necessity for such uniformity was more marked than in the analysis of brewing materials.

Mr. HEHNER said that there were many articles besides beer in the case of which the chemist was called upon to make analyses of products which were afterwards used by the manufacturer; and the question arose as to what the manufacturer expected to learn from the analyses. Did he expect to learn how much the manufacturer's yield would be? or the actual quantity of any given constituent present in the article examined? In a gold ore, for example, the manufacturer desired to know how much gold was actually present; whether he got out that amount was another question, and probably in no two cases would the quantity turned out be the same. He (Mr. Hehner) himself made a great many analyses of crude glycerine, which was afterwards worked up into pure glycerine by distillation. He determined the actual percentage of glycerol present in the samples, not the quantity a manufacturer would expect to get out, which varied very widely. His function was to educate the manufacturer into a knowledge of what theoretically he might get out, it being the manufacturer's business to work as near to theory as possible. The same was presumably the case with malt. The chemist ought to say how much extract the brewer, with the most careful working, should be able to obtain. The brewer would understand that on a large scale there must be a certain loss, and that he would not quite reach the ideal.

Mr. LING said that if it were possible to estimate the starch in malt with anything like precision, a valuable scientific figure would be the extract calculated from the percentage of starch. The latter was the main extract-yielding constituent of the malt.

Mr. BRIANT, in reply, said that for many years he had made use of the process described by Mr. Ling. It was very good in its way, but there was still the same error arising from the solution of the sugar, which was not overcome any more than in the ordinary process of Mr. Heron. If merely the theoretical extract was to be given, then, as Mr. Ling had said, the starch could not be made the basis of calculation—at any rate, until a good method was devised for estimating it. If the theoretical extract was to be given, he thought it would be wise to adopt the recommendation of one of the Continental conferences—namely, that the whole of the malt should be ground to an extremely fine powder, and that the water should be at first cold, and gradually raised practically to boiling-point. Then everything extractible would be extracted, and something approaching uniformity might be hoped for between different chemists. But he doubted whether a figure so obtained would be appreciated by the brewer. He agreed to a certain extent with Mr. Hehner; but the brewer desired to get from his chemist some idea of the quantity of extract he was likely to obtain, perhaps by making a certain deduction from the figures of the chemist. Sometimes, for particular classes of beer, the brewer knowingly sacrificed extract, or, in other words, mashed at a temperature at which he knew the maximum extract would not be yielded. It paid him to do so in order to get quality. But he looked to his chemist for an approximate figure; and if a satisfactory figure of that kind was to be obtained, the endeavour should be made to eliminate an error such as occurred in all those processes in which no proper allowance was made for the difference in the quantities of husk in different samples of malt. This difference was considerable, and unless allowance was made for it, undoubtedly an error was introduced into the analyses. No doubt, as Mr. Chapman had said, the absolute amount of extract could not be given which a brewer could obtain; but a comparative figure could be given, and the brewer would in practice be able to decide how nearly that figure approximated to his own result.

THE DETECTION OF ARSENIC IN BEER AND IN BREWING MATERIALS.

By Alfred C. Chapman, F.I.C.

That the arsenic which has recently been detected in samples of beer was introduced through the medium of glucose or invert-sugar, in the manufacture of which arsenical sulphuric acid had been used, is now certain. Both to the private practitioner and to the public analyst this discovery is resulting in a very considerable influx of work; and it has been suggested to me that a brief account of the very extensive experience I have had during the past week or two in connection with the examination of beer and brewing materials for traces of arsenic might not be without interest to those members of our society who are now being called upon to examine such samples in connection with the Sale of Food and Drugs Act. To every chemist confronted with a problem such as this, the methods of Marsh and of Reinsch would naturally at once suggest themselves. Other methods, such as those of Fleitmann and of Gutzeit, in their original or modified forms are excellent in certain circumstances, and for purposes of obtaining additional confirmation perhaps, but they suffer under the great disadvantage of not yielding any characteristic and recognisable arsenic compound of a permanent and tangible nature—something, in fact, which can be preserved and brought forward as evidence should any dispute occur. I do not, indeed, think that any chemist would care to rely solely upon any method other than that of Marsh or of Reinsch in work of this character, and in cases which might give rise to legal proceedings, and which might ruin the reputation of a manufacturer. Of these two methods, that of Marsh is undoubtedly the more sensitive, provided that the arsenic in the organic matter to be dealt with can be got into a suitable form. This preliminary treatment is troublesome and takes much time, placing the process at a serious disadvantage as compared with that of Reinsch, where hundreds of samples have to be dealt with in a comparatively short time. If, on the other hand, an attempt is made to utilize Marsh's method working directly on the beer or sugar, then I am of opinion that not only is Reinsch's more convenient, but that it is at least equally sensitive. I have, in fact, on several occasions obtained much more distinct evidence of the presence of traces of arsenic in certain sugar materials by the latter than by the former, using in both cases simply solutions of the sugar itself.

The following is the procedure I have adopted for the detection of traces of arsenic in beer:

From 300 c.c. to 500 c.c. of the beer are transferred to a beaker, acidified with a few drops of hydrochloric acid, and heated on a sand-bath until the bulk of the alcohol has been driven off. A quantity of pure hydrochloric acid, roughly equal to one-fifth of the remaining volume of beer, is then added, and a small piece of copper-gauze, about [illegible] inch × [illegible] inch, attached to a copper wire, is then introduced. If at the end of one hour's gentle boiling the copper has remained quite bright, it may safely be asserted that the beer was free from arsenic, or contained less than 1 part per 1,000,000 of arsenious acid. Should the copper be at all discoloured, however, it is removed from the beaker, thoroughly washed first in a stream of running water, then with alcohol, and finally with ether, after which it is dried thoroughly by gently warming it several inches above a small flame, and is carefully folded and introduced into a glass tube of small section, closed at one end, and about 3 inches long.

On gradually heating the end of this tube to redness (the tube being held in a nearly horizontal position), any arsenic present will become oxidized, and a crystalline sublimate in the form of a ring will be obtained. This, which has a very characteristic appearance to the trained eye, should then be examined by means of a ¼-inch or ½-inch objective for the presence of the well-known crystal-forms assumed by arsenious oxide under these conditions.

In the case of heavy traces of arsenic, a ½-inch objective is sufficient; but for the sublimate which corresponds with a minute trace a ¼-inch objective is necessary. It is, of course, well known that under these circumstances perfect octohedra are comparatively rare, the majority of the crystals being modified forms of the octohedron or tetrahedra. In the case of very faint sublimates the crystals have, as a rule, the appearance of minute triangles. Sulphites of various kinds are often added in small quantity to beer as preservatives, and when present will, of course, give rise to the formation of black copper sulphide, should any trace remain after the boiling above referred to before the insertion of the copper. This stain, however, remains unchanged when the copper is heated in the small tube, and, of course, gives no sublimate. Occasionally, too, a slight film of organic matter diminishes the brightness of the copper, but this also could not possibly be mistaken for arsenic.

Experimenting with samples of beer which gave not the slightest reaction for arsenic, I have found it quite possible to detect with certainty 1 part of arsenious acid in 1,000,000 parts, and have had no difficulty whatever in obtaining a well-formed crystalline sublimate with that quantity working with 400 to 500 c.c. of the beer. The copper gauze I have used is the finest obtainable, and it should be well washed with alcohol and then with ether before being used. Should it be at all tarnished when purchased, the alcohol and ether washings should be preceded by treatment with dilute nitric acid, as a brilliant metallic surface, free from grease, is essential to the detection of very minute traces.

In the case of glucoses, invert-sugars, and similar materials, I have worked on 50 grammes dissolved in 200 c.c. of water, and acidified with 50 c.c. of hydrochloric acid. Some of these substances carbonize considerably during the latter part of the hour's boiling, and it will be found very desirable to remove the copper at intervals of, say, ten minutes for inspection. Should a distinct discoloration occur during the first ten or fifteen minutes, the copper should then be removed and examined as above. In this way it is frequently possible to obtain an indication before the separation of carbon, which is apt to adhere rather strongly to the copper, and to so spoil the clearness of the crystalline sublimate. I may add that, when operating in the above manner, I have had no difficulty in detecting traces of arsenic when present as *arsenic* acid.

Carried out as above suggested, Reinsch's method will, I am sure, fully answer the requirements of public analysts when dealing with such materials as I have referred to, combining as it does ease and rapidity of execution with certainty of indication and sensitiveness. In addition to beers which were distinctly arsenical, and in which impure glucose or invert-sugar had been used, I have met with a considerable number containing *very minute traces* of arsenic, and in which it was within my knowledge that malt alone had been employed. On examining samples of malt, I discovered that, when working on 100 grammes, a considerable number gave reactions indicating the presence of an almost infinitesimal trace of arsenic; and in one or two instances I have been able clearly to demonstrate the connection between the presence of this exceedingly minute trace of arsenic in the malt and that present in the beer.

Whether this is due to the coal used for kilning, or whether it may not in some cases be referred back to the barley, remains to be shown; but it is obvious that the presence of very minute traces of so widely distributed an element as arsenic must be interpreted with caution. Its existence, for example, in wine containing no artificial colouring matter has already been recorded; and it is quite possible that it may be detected in a number of hitherto unsuspected natural products now that attention has been drawn to the matter in so striking a manner. The very minute traces I have above referred to give reactions which cannot for a moment be confounded with those yielded by beer which has been brewed or primed with arsenical sugar.

[Since the above communication was made to the society, the committee of experts appointed by the Manchester Brewers' Central Association has recommended a method for the detection of arsenic in beer identical in all essentials with that described.—A. C. C.]

DETECTION OF ARSENIC IN BEER.

By ALFRED H. ALLEN.

THE recent correspondence in the papers, emanating chiefly from chemists who have been concerned in the deplorable epidemic of arsenical poisoning in Lancashire, has led to some interesting disclosures. Especially curious are the statements made respecting the delicacy of Marsh's test for arsenic, and its suitability for examining beer for that element.

Mr. William Kirkby states that it is doubtful whether Marsh's test, under the most favourable conditions, will indicate less than 1 grain of arsenic in 100,000 parts, or, say, about three-quarters of a grain per gallon of beer. This astounding experience may be contrasted with the statement of Mr. Frank Scudder—apparently rather on the dubious authority of the text-books than as the result of his personal experience—that a mirror on a porcelain basin in which you can see the reflection of your face can be obtained from one-forty-fifth of a gallon of a liquid containing 1 part of arsenious oxide in 700,000. But Mr. Scudder does not in practice employ these quantities, preferring to operate on not less than 1 litre of the beer.

Mr. William Thomson finds that, working on 150 c.c. of the beer, an amount of arsenic equal to one-hundredth of a grain of As_2O_3 can be detected with certainty by Marsh's test. He attributes the difficulty many analysts experience in detecting arsenic in beer to the imperfect or unsuitable methods they adopt to destroy the organic constituents of the liquid.

Mr. Chas. Estcourt has done good service by reminding analysts of the frequent presence of sulphites in beer, and of the fact that such beer will not give an arsenical mirror with Marsh's test. It is well known that sulphites evolve sulphuretted hydrogen when treated with zinc and dilute acid, and if this is produced in sufficient amount it may cause complete conversion of the arsenic into sulphide in the evolution-flask, and so remove it from the sphere of action. But I have in mind a case in which the material (burnt pyrites), when examined by Marsh's test, gave a yellow deposit of arsenious sulphide just beyond the heated part of the tube. At any rate, it is clear that sulphites interfere with the employment of Marsh's test, and should be previously removed. Mr. Estcourt effects this by boiling the beer for some time with acid before adding the metal. This appears to me an uncertain and tedious method, and I effect its immediate destruction by adding an oxidising agent, such as hydrogen peroxide, bromine-water, or potassium permanganate, to the acidulated beer. I prefer bromine-water, as it does not act on the organic matter so readily as permanganate, and the excess can be readily removed by a few minutes' boiling. Whichever plan be adopted, the arsenic is oxidised to the arsenic state, and some time elapses before the liquid will evolve arsenical hydrogen in Marsh's apparatus. This inconvenience I avoid by adding a few drops of a solution of cuprous chloride in hydrochloric acid to the contents of the evolution-flask. This reagent instantly reduces the arsenic to the arsenious state, and arsenical hydrogen is evolved almost immediately.* The use of cuprous chloride has the additional advantage that it results in the formation of a copper-zinc couple, and the evolution of hydrogen is much facilitated.

A working difficulty in the examination of beer by Marsh's test is met with in the persistent frothing of the liquid. This may be in a great measure overcome by previously boiling the beer till it is reduced to about half its original bulk. Even then a capacious flask is necessary. Oxidation of the organic matter also mitigates the difficulty.

Mr. W. Kirkby has described his method of applying Gutzeit's well-known mercuric chloride test for arsenic. He employs 3 c.c. of the beer, which is a proof of the delicacy of the test, and apparently its sole advantage. A yellow stain on a piece of filter-paper does not appeal to my mind in the same way as an arsenical mirror or the formation of crystals of characteristic form. Messrs. Paul and Cownley fully endorse this view, and consider that to obtain a satisfactory result with the mercuric chloride test is quite out of the question.

Messrs. A. Wynter Blyth and H. G. Madan independently recommend the treatment of beer with caustic alkali and aluminium, and utilise the reaction of the evolved hydrogen with silver nitrate as a proof of the presence of arsenic. The method is very simple, and eliminates interference by the presence of sulphites or antimony; but the production of a black stain or precipitate does not appear to me so convincing as the formation of crystals.

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* A well-known analytical chemist informs me that, in his experience, the addition of cuprous chloride prevents the formation of arsenuretted hydrogen, but a careful retrial of my suggestion does not enable me to confirm this observation.

I have recently had a considerable number of samples of beer to examine for arsenic, and find Reinsch's test to be the most satisfactory. The hydrochloric acid employed is purified by distilling off about one-tenth, this fraction containing the minute trace of arsenic originally present. I operate, as a rule, on 100 c.c. of the beer, and, as a preliminary treatment to eliminate sulphites, add hydrochloric acid and a little bromine-water, and boil the liquid for a few minutes. To obviate the difficulty caused by the fact that arsenic acid only responds to Reinsch's test after prolonged boiling and in presence of much acid, I next add a little solution of cuprous chloride in hydrochloric acid, which reduces the arsenic to the arsenious condition. On now introducing about 1 square centimetre of copper-foil and boiling, any arsenic is promptly deposited on the copper. The boiling is continued for thirty minutes, replacing any water lost by evaporation.* If the copper has then undergone darkening, it is dried in the water-oven, cut into strips, and heated in a narrow tube, when a characteristic deposit of arsenious oxide, in the form of microscopic octohedra or tetrahedra, will be obtained if the deposit on the copper was due to arsenic. Unless these crystals can be obtained, I am not satisfied that arsenic is present. The definition of the crystals is improved by filling the sublimation-tube with water, which acts only very slowly on crystallized arsenious oxide, and is preferable to alcohol.

This process has the advantage that the arsenic is actually seen and identified as such. Operating as above described, one part of As_2O_3 in 2,000,000 of beer gives a perfectly distinct reaction; but the delicacy of the test can be further increased by employing a larger quantity of the beer. Several sublimates can be united, and the arsenic again deposited on copper or subjected to Marsh's test.

The expert committee appointed by the Manchester Brewers' Central Association have issued a report in which they recommend Reinsch's test as the best for the detection of arsenic in beer. Unfortunately, they make no mention of the importance of insuring that the arsenic is in the arsenious condition. This omission has resulted in a reminder from Mr. J. A. Wanklyn that in 1861 an eminent German chemist (name not stated) made an elaborate investigation, which showed that 20 grains of arsenic "in one of its commonest forms" (form not stated in the original letter, but subsequently acknowledged to be arsenic acid), dissolved in a gallon of liquid, would remain undetected by the method recommended by the experts, or even on continuing the boiling for forty-five hours. A valuable detail in the experts' report is the direction to warm the upper part of the sublimation-tube before heating the copper. This precaution results in the deposition of larger crystals of arsenious oxide than can otherwise be obtained. It seems highly probable that when deposition of arsenic from an arseniate occurs in Reinsch's test, cuprous chloride has been previously formed by the action of the hydrochloric acid and atmospheric oxygen on the metallic copper, and that in the complete absence of air a negative reaction would be obtained.

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* Dr. Dupré insists that the boiling should be conducted in a flask furnished with a reflux condenser, while other operators employ an open basin and encourage concentration by evaporation.

P.S.—Mr. A. E. Berry states that, in many cases where the deposit obtained in Marsh's test very closely resembled arsenic, on further investigation he found it to consist chiefly of "sulphur compounds." He suggests that "oxidised sulphites" are reduced to sulphuretted hydrogen by the nascent hydrogen produced in Marsh's test. "Oxidised sulphites" can only mean sulphates, and if these are reduced as alleged, a deposit of "sulphur compounds" would always be met with in applying Marsh's test.

ARSENIC IN BEER.

REPORT OF THE COMMISSION TO THE MANCHESTER BREWERS' CENTRAL ASSOCIATION.

GENTLEMEN,

The Commission have now examined the sugars and all the other materials used in brewing in Manchester (with the exception of the malt), and they consider that it is clearly established that the arsenic found in deleterious quantities in the beer has been solely due to the contamination by arsenic of the sugars supplied by Messrs. Bostock and Company, Limited, of Liverpool. The arsenic in these sugars was undoubtedly derived from the impure sulphuric acid used in their manufacture.

The measures recommended by the Commission a few days ago have, they learn, been effectively carried out. The whole of the beer in the manufacture of which any Bostock sugar was used has been destroyed, and there is no further danger from this source.

All the other brewing sugars on the English market have been analysed, and have been found to be quite free from arsenic.

In view of the importance of the matter, the Commission have instituted inquiries as to the manufacture of brewing sugars in the United Kingdom, and in this they have been assisted by the whole of the manufacturers of such sugars, in a body, voluntarily offering them the opportunity of examining into the mode in which they conduct the manufacture. These manufacturers have stated collectively that it is, and always has been, their custom to use only acid free from arsenic, and they have requested the Commission to examine their invoices, works, etc., in order to verify these statements. Lack of time has prevented this being done by personal examination up to the present, but the fact that all the specimens of brewing sugars, as well as of glucose used for other purposes, on the market have been found to be free from arsenic, and the absence of any previous cases in which arsenic has been in such products leave in the mind of the Commission no doubt that this statement, when examined into, will be found to be correct.

The Commission are quite unable to explain how Messrs. Bostock and Co., Limited, came to employ an acid of the character actually used by them. The absolute necessity of using an acid free from arsenic in the manufacture of an article for human consumption must have been evident to everybody technically connected with the manufacture, and such acid is a common article of commerce. The price of the sugars in question was as high as any in the market, and, apart from this, the quantity of acid used in the manufacture of the sugars is so small that the difference in cost of the best and the worst would only make a difference of a fraction of a penny per hundredweight of sugar. So that it is not a case of an attempt to cheapen production by the use of lower priced materials. The Commission believe that it is this inexplicability which has rendered the matter so serious, and that the extent to which the mischief spread before it was detected was mainly due to the fact that the use of an acid containing arsenic in the manufacture of sugars was a contingency so improbable that it never occurred to those purchasing the sugars that it was possible that any danger could arise from that quarter.

The Commission have been unable to detect the presence of arsenic in brewing materials other than sugar supplied by Messrs. Bostock and Company, Limited; but they are aware that it has been asserted that traces of arsenic have been found in various samples of malt and hops. If such traces exist, they have probably been introduced in the operation of kilning, and the Commission propose to examine more fully into the matter. None of the specimens of hops have as yet yielded any traces. In any case, the amount so introduced would appear to be exceedingly minute, and not sufficient to have any deleterious effect.

The most important matter for the moment is to secure the adequate testing of beer, in order that the public may be protected from all further mischief. Arsenic is a substance which can be detected in the most infinitesimal quantities by those who are practised in the test; but these tests are so delicate that they are apt to mislead those who have not had experience in their application, and this is more particularly the case when the test is to be applied to a complex substance such as beer. Accordingly, the Commission have thought it necessary to investigate, and determine what is the most suitable method of testing beers for arsenic.

TEST.

The Commission recommend that the Reinsch test should be used in preference to all others at present known, because their investigations have satisfied them that it is the best and most reliable test for arsenic in beer. The mode of performing it is as follows:

> Take 200 c.c. of the beer in a porcelain evaporating dish. Raise the liquid to the boiling-point, and then add 30 c.c. of pure concentrated hydrochloric acid. Insert a piece of pure bright copper foil, about ¼ inch by ½ inch in size, and keep the solution gently boiling for forty-five minutes. If at the end of that time the copper remains bright and red, the beer is free from arsenic.

If a deposit is obtained on the copper, the foil is to be washed successively with water, alcohol, and ether (care being taken that these are pure), dried at a temperature not exceeding 100° C., and subjected to slow sublimation in a thin reduction-tube of small section, and not less than 2 inches long, the upper portion of which should be warmed before the sublimation begins. For the purpose of the sublimation, a small spirit-lamp flame should be used. If any sublimate is obtained, it must be examined under a magnifying power of about 200 diameters. Any sublimate which does not show well-defined octahedral or tetrahedral crystals is not to be considered arsenical.

> N.B.—It must be borne in mind that the blackening of the copper or a deposit thereon from the preliminary operation does not demonstrate the presence of arsenic in beer. Abundant blackening and deposit may be obtained from the purest beer.

Lauder Brunton,
Thos. Stevenson,
Alfred Gordon Salamon,
Arthur P. Luff,
Samuel Buckley,
J. Fletcher Moulton.

December 15, 1900.

SOIL SAMPLING.

Erratum.—In the report of Dr. Dyer's remarks on the sampling of soils, in the discussion of Mr. A. D. Hall's paper "On Uniformity in Soil Analysis" (p. 287), the following sentence occurs: "He (Dr. Dyer) had recently had a conversation with Sir Henry Gilbert on this subject, and learnt that at Rothamsted the view now held was to the effect that a more suitable depth would be one-fifth of a metre, viz., something under 6 inches—a convenient international measure." "6 inches" was a misprint for "8 inches."

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

On the Chemical Action of the Growth of Moulds upon Butter. J. Hanus and A. Stocky. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1900, iii., 606-614.)—The authors have studied the chemical changes produced in butter by the growth of various moulds. The butter was used in the ordinary state in which it comes on the market, and for the purpose of the experiments was spread upon glass plates in thin layers. The moulds used were pure cultures. Preliminary experiments extending over three months were made with a variety of different moulds, and from the experience thus obtained, a further and more extended experiment was undertaken with the *Mucor mucedo*. The mould was allowed to vegetate for twelve months, the butter being examined before and after, and the results compared. The principal change noticeable was a great increase in the acid number, the acidity of the butter being augmented about twentyfold by the growth of the mould, and it would appear that these organisms have the power of resolving the glycerides into their constituent parts. At the same time, a secondary change, consisting in the production of bodies of an aldehydic character, was observed; but this the authors are not yet in a position to discuss. H. H. B. S.

Detection of Gelatin in Fruit-Jellies. O. Henzold. (*Zeits. öffentl. Chem.*, 1900, vi., 292; through *Chem. Zeit. Rep.*, 1900, 260.)—The sample is diluted with water, boiled, and, if necessary, filtered. It is then mixed with excess of a 10 per cent. solution of potassium bichromate, brought to the boil, and cooled immediately by placing the tube in cold water. When cold, 2 or 3 drops (carefully avoiding more) of strong sulphuric acid are added. In presence of gelatin a precipitate appears, at first white and flocculent, soon cohering into lumps, and settling to the bottom of the liquid. No gelatinous substances derived from plants respond to this test, but hitherto it has not proved available for the quantitative determination of gelatin.

F. H. L.

Detection of Saccharin, Salicylic Acid, and Mixtures thereof. E. Riegler. (*Pharm. C. H.*, 1900, xli., 563; through *Chem. Zeit. Rep.*, 1900, 291.)—The reagent is a solution of *p*-diazonitraniline, prepared by dissolving 2·5 grammes of *p*-nitraniline in 25 c.c. of water and 5 c.c. of strong sulphuric acid, diluting with 25 c.c. of water, agitating with a solution of 1·5 grammes of sodium nitrite in 20 c.c. of water, and finally diluting to 150 c.c. The liquid is permanent if kept in the dark.

To detect saccharin, 0·01 to 0·02 gramme of the sample is dissolved in 10 c.c. of water, mixed with 2 drops of 10 per cent. sodium hydroxide, and poured into a 30 c.c. stoppered separating funnel. The reagent is added drop by drop with agitation till the green-yellow colour of the liquid disappears (about 10 drops are required). Ten c.c. of ether are next introduced, the whole is well shaken, the aqueous portion is run off, and the ether is treated with 20 or 30 drops of 10 per cent. sodium hydroxide. After agitating for half a minute, the liquid is allowed to separate, yielding a yellowish-brown aqueous portion and a green ethereal one. On running off the former and adding 5 c.c. of ammonia to the ether, it is decolorized, the ammoniacal liquor becoming blue-green.

To detect salicylic acid the manipulation is the same. The aqueous liquid is bright red, the ether colourless. After treatment with ammonia the ether is colourless, the aqueous portion red. To detect mixtures 0·02 to 0·03 gramme of the sample are taken and treated similarly. The ether is green, the water red. Ammonia decolorizes the former and itself becomes violet. The violet colour alters in shade according to the proportions of the two substances present.

F. H. L.

Composition and Determination of Cerium Oxalate. F. B. Power and F. Shedden. (*Journ. Soc. Chem. Ind.*, 1900, 636.)—The commercial salt employed for medicinal purposes contains about 40 per cent. of cerium oxalate, the remaining 60 per cent. consisting mostly of the oxalates of lanthanum and didymium. The authors find that these substances all have the general formula—$R_2'''(C_2O_4)_3 \cdot 10H_2O$.

The following qualitative test for cerium, due to Knorre, is extremely delicate: A gramme of the substance is heated with half a c.c. of sulphuric acid, 10 c.c. of water are added and a gramme of ammonium persulphate. On heating the liquid to boiling, a bright yellow colour is produced if cerium be present.

The most accurate method for estimating cerium in the presence of lanthanum and didymium is based on the same principle (see ANALYST, 1898, 191). The cerium is oxidized to the ceric state by means of persulphate, and then the quantity of standard reducing solution required to reconvert it into the cerous condition is determined. Half a gramme of the oxalate is gently heated with 1 c.c. of pure sulphuric acid. When all effervescence has ceased, 100 c.c. of water are added. The solution should now be perfectly clear. If any precipitate separates out, it must be dissolved by the addition of more acid. A solution is made of 4 grammes of ammonium persulphate in 20 c.c. of cold water. The cerium solution is cooled somewhat below 50° C., 10 c.c. of the persulphate solution are added, and the liquid is again boiled for a minute or two. At this stage a precipitate often separates out, but it disappears as the oxidation proceeds. The remainder of the persulphate solution is added in exactly the same way in two separate lots of 5 c.c. each, the final boiling being prolonged to fifteen minutes. Four c.c. of sulphuric acid are now diluted with 20 c.c. of water, and added to the hot liquid. It is important to avoid the use of too much acid; otherwise the ceric salt may be reduced through the formation of hydrogen peroxide. The solution is now reduced to room temperature, and diluted to 100 c.c. Decinormal solution of ferrous sulphate is now run in until the deep yellow colour is entirely discharged. The excess of ferrous sulphate is determined by running in decinormal permanganate solution until the pink tint remains permanent for more than a few seconds. The number of c.c. of decinormal ferrous sulphate solution required per gramme of substance taken, multiplied by 1·40, gives the percentage of cerium.

Fairly accurate results may also be obtained by digesting the ignited oxides with potassium iodide and hydrochloric acid, and determining the iodine liberated. The presence of praseodymium makes the results high. The ignited oxides from half a gramme of oxalate are placed in a 100 c.c. stoppered bottle, with a gramme of potassium iodide and 5 c.c. of hydrochloric acid. If the oxides are brown, showing the presence of didymium, 5 c.c. of water are also added. The air is displaced by carbon dioxide, and the bottle is placed on the top of a steam-oven, and rotated from time to time for about half an hour until solution is complete. When the bottle is cool, 50 c.c. of water are added, and the solution is titrated with decinormal thiosulphate solution in the usual way.

In making a complete examination, determinations may also be made of the oxalic acid by titration with permanganate solution, and of the residue left on ignition, which should consist almost entirely of the oxides of cerium, lanthanum and didymium.

A. M.

TOXICOLOGICAL ANALYSIS.

Note on a Method of detecting Free Phosphorus. P. Mukerji. (*Chem. News*, lxxxii., 205.)—The method depends on the phosphorescence of phosphorus vapour diluted with hydrogen. The apparatus used consisted of a three-necked Woulffe's bottle; a safety funnel dipping into the liquid in the bottle is fitted in the neck, a short jet, and a wide tube, about 11 inches long, and closed by a cork, being inserted in the other two, so as to enter the bottle for a short distance only. The wide tube is loosely fitted to allow traces of air to enter the bottle. To carry out the test, zinc and dilute sulphuric acid are placed in the bottle. When the mixture has become hot, the substance to be examined is introduced, and the appearance of the issuing gas observed in a dark room. If phosphorus is present, it will glow. If the cork is then taken out of the wide tube, the glow will sink through the jet and appear at the top of the wide tube, but it reappears at the jet if the cork is replaced.

The test is at least as delicate as that of Mitscherlich; 2 milligrammes of phosphorus could be detected in the presence of 112 grammes zinc, 250 c.c. dilute sulphuric acid, and 30 c.c. milk. By passing the vapour through silver nitrate solution, the phosphorus may be estimated quantitatively. Many substances which interfere with the detection of phosphorus by other methods, especially phosphorous compounds, do not affect this one.

Before opening the bottle used for the experiment, it should be filled with water.

A. G. L.

Detection of Digitalis in Cases of Poisoning. D. Vitali. (*Boll. Chim. Farm.*, 1900, xxxix., 597; through *Chem. Zeit. Rep.*, 1900, 301.)—Of the various constituents of digitalis, digitoxin exists in the largest proportion, and seems to be the body which is best sought for in cases of poisoning. To test the possibility of detecting it, a mixture was prepared of 200 grammes of chopped horseflesh, 100 grammes of urine, and 3 grammes of powdered digitalis. This was macerated for twenty-four hours, evaporated to dryness on the water-bath, and the residue extracted twice with 70 per cent. alcohol. The liquid was filtered, the spirit distilled off, the aqueous portion concentrated to half its bulk, and precipitated with lead acetate, removing the excess with sodium sulphate as usual. The filtrate was made alkaline with ammonia, and extracted with chloroform. On evaporation of the latter, a pale residue was left, which was again taken up in chloroform and treated with a mixture of 1 part of ether and 7 parts of petroleum spirit to throw down the digitoxin. The precipitate was dissolved in alcohol, evaporated, and taken up in ether. The solid matter contained in the ether was scarcely visible, but it gave the Keller test: It was dissolved in 2 c.c. of a solution of 1 part of ferric chloride in 500 parts of glacial acetic acid, and floated on strong sulphuric acid, when a red-brown colour appeared between the liquids, which afterwards changed to blue. In the aqueous liquid left after extraction with chloroform, digitonin and digitalin ought to be found, but the tests failed, and they are probably only capable of being recognised when large doses of digitalis have been administered.

F. H. L.

ORGANIC ANALYSIS.

Modification of Ritthausen's Process for determining Albumin. F. Barnstein. (*Landw. Versuchsst.*, 1900, liv., 327; through *Chem. Zeit. Rep.*, 1900, 313.)—Instead of endeavouring to throw down the precipitate of copper oxide and albumin by completely neutralizing the liquid with sodium hydroxide, the present author prefers to add a measured quantity of alkali insufficient to throw the whole of the copper out of solution. In this way the difficulty of exactly neutralizing a coloured liquid is avoided, the solutions are more easily prepared and keep better, and the use of alum, which is inconvenient with substances containing much potassium phosphate, is not necessary. One or 2 grammes of the sample are boiled, or heated for ten minutes on the water-bath, with 50 c.c. of water, and then mixed with 25 c.c. of copper sulphate solution containing 60 grammes of crystals per litre. Twenty-five c.c. of caustic soda (12·5 : 1,000) are next introduced, and the whole is allowed to settle. The precipitate is washed by decantation, then brought on a filter, and washed till free from copper and sulphuric acid. The nitrogen in it is finally estimated by the Kjeldahl process. The results agree well with those obtained by the Stutzer method. In substances such as tea, tobacco, etc., which contain alkaloids or other nitrogenous constituents, however, rather more nitrogen is recovered; but alkaloids, etc., never occur in sufficient quantity in food-stuffs to render a distinction between them and the albumin requisite. Barnstein's process does not throw down peptones entirely, but the basic copper precipitate of either vegetable or animal albumin always contains more nitrogen than Stutzer's copper hydroxide precipitate, as might be expected. F. H. L.

Preparation of Paper for Microscopic Examination. W. Herzberg. (*Mitth. Kgl. techn. Versuchsanstalten*, Berlin, 1900, xviii., 86; through *Chem. Zeit. Rep.*, 1900, 254.)—In order to obtain the fibres in a fit state for microscopic examination, unsized paper—blotting, filter, and "half-stuff," etc.—is disintegrated by boiling in water; and materials containing wool are treated similarly. All other varieties are boiled with 4 per cent. sodium hydroxide, cardboard and the like being first split into thin sheets, to allow the alkali to penetrate more thoroughly. The pulp, which is already of a yellow-ochre colour from the action of the soda if wood-fibre is present, is strained through a fine sieve, and washed with water. It is next brought into a wide-necked stoppered flask, half full of water and containing some small pebbles; and the whole is shaken till all lumps are broken down. The product is filtered, and is then ready for examination under a power of 150 diameters. Two stains may be employed to assist in the identification of the various fibres: (A) potassium iodide, 2 grammes; iodine, 1·15 grammes; glycerin, 2 c.c.; water, 20 c.c.; (B) zinc chloride, 20 grammes; water, 10 c.c.; potassium iodide, 2·1 grammes; iodine, 0·1 gramme; water, 5 c.c.: mixed together, clarified by subsidence, and treated with a crystal of iodine. The effects are as follows:

	A.	B.
Linen, hemp, cotton	Black to dark brown; thin scales almost colourless.	Faint to strong wine red.
Wood-cellulose, Adamsonia ...	Gray to brown.	Blue to red-violet.
Straw-cellulose, jute	Gray.	Blue to blue-violet.
Esparto	Partly gray, partly brown.	Partly blue, partly wine red.
Manila	In parts gray, brown, and yellow-brown.	Blue, blue-violet, red-violet, dirty yellow, and greenish-yellow.
Wood-powder, raw jute, etc. ...	Yellow-brown and yellow, according to thickness.	Lemon to dark yellow.
Straw-stuff	Yellow-brown, yellow, gray.	Yellow, blue, blue-violet.

F. H. L.

Estimation of Starch in Potatoes. G. Baumert and H. Bode. (*Zeit. angew. Chem.*, 1900, 1074 and 1111.)—The authors have devised a method for determining directly the amount of true starch in potatoes. The cellulose is first removed by digesting in an autoclave and filtering. The starch is then separated from nitrogenous and other substances, by precipitation with alcohol in alkaline solution. It is thus obtained mixed only with a little inorganic matter. The weight of starch is found by determining the loss on ignition.

Of the finely-ground air-dry material, 3 grammes are weighed out and stirred with 50 c.c. of water in a porcelain beaker. This is allowed to stand about half an hour, with occasional stirring; then the liquid is decanted off through a small asbestos filter. The filter is put back in the beaker, 50 c.c. of water are added, the beaker is covered and is heated for three and a half hours in a Soxhlet autoclave at about 3 atmospheres pressure (not more). When cool, the contents are transferred into a 250 c.c. flask, washing in with boiling water. This is then boiled for ten minutes, allowed to cool, and filled up to the mark. It is thoroughly mixed, 100 c.c. are filtered off, and 10 c.c. of 10 per cent. caustic soda solution are added to the filtrate, whereon the opalescent liquid becomes clear. Twenty-five c.c. of this and 100 c.c. of alcohol (94 to 96 per cent.) are stirred together in a beaker; before the precipitate collects together about a gramme of finely-divided asbestos is added, and the liquid is stirred vigorously until the precipitate settles quickly, leaving the solution clear. If this does not occur in about a minute, more asbestos should be added. The liquid is now decanted on to an ignited asbestos filter, and drawn through. The precipitate is stirred with 80 per cent. alcohol, brought on to the filter and washed, taking care not to press the precipitate or to allow it to become dry. The beaker is washed round with 3 to 5 c.c. of 5 per cent. hydrochloric acid, 25 to 30 c.c. of alcohol are added to reprecipitate starch, and this is poured on to the filter, which is now washed first with 80 per cent. alcohol, then with absolute alcohol, and finally with dry ether. The filter-tube is then dried to constant weight at 120° to 130°, with a current of air passing through it. After weighing, the tube is ignited with a current of air or oxygen passing through, and then again weighed. The loss of weight is the quantity of true starch in $\frac{3}{11}$ gramme of the air-dry material.

The method gives concordant results, which agree with those obtained by inverting and estimating the starch as dextrose. In a sample of purified starch, 85·39 per cent. was found, as against 85·17 per cent. estimated by difference after determining the moisture and impurities. A. M.

Digestion and Assimilation of Pentoses and Furfuroids. **C. F. Cross, E. J. Bevan, and J. S. Remington.** (*Journ. Amer. Chem. Soc.*, 1900, 630.)—The authors have performed digestion experiments with the furfuroid-yielding constituents of plant tissues, which are generally termed pentosans, but which the authors prefer to call furfuroids. These were extracted from brewers' grains by digestion with 1 per cent. sulphuric acid at 130° C., and purified by neutralizing, filtering, evaporating. The extract when tested by distillation with hydrochloric acid gave 3·95 per cent. of furfurol, calculated on the dry weight. It was found that the product, mixed with gelatin, bread, and fresh vegetable food, was practically entirely assimilated when administered to rabbits. A. M.

Digestibility of some Non-nitrogenous Constituents of Certain Feeding Stuffs. **G. S. Fraps.** (*Journ. Amer. Chem. Soc.*, vol. xxii. [9], pp. 543-552.)—The conclusions drawn by the author from the experiments detailed in the paper are that the sugars in feeding stuffs are, as a rule, completely digested, and their determination is of special importance in the case of hay and cotton-seed meal. The average digestibility of pentosans (thirty-four samples) is 64·2, or in eight samples of timothy hay 53·9. The order of digestibility of the non-nitrogenous extractive matter is (1) sugars, (2) starch, (3) pentosans, (4) residue. The pseudopentosans of crude fibre are less digestible than the residue, whilst the latter is sometimes more digestible, in other cases less so, than the residual non-nitrogenous extract. Crude fibre may undergo such alteration in the digestive process as to appear as non-nitrogenous extract in the excrement, and its estimation is less important than that of sugar, starch, and pentosans. C. S.

Extraction of Fat from Animal Organs. **G. Rosenfeld.** (*Centralb. inn. Med.*, 1900, xxi., 833; through *Chem. Zeit. Rep.*, 1900, 250.)—It is well known that mere extraction with ether does not suffice to recover all the fat from animal organs. Pflüger and Dormeyer have suggested submitting the materials to a previous peptic digestion, and then treating with ether; but the process is tedious and complicated. By extracting the organs for six hours with chloroform in a Soxhlet, the results are similar to those of the Dormeyer process; but still higher figures are obtained if they are first boiled for a short time with alcohol. The final solutions are evaporated, the residue placed for a time in a drying oven, then dried thoroughly in the desiccator, taken up once more in cold absolute ether, evaporated and dried again, thus insuring that only substances really soluble in ether are weighed. Nevertheless, the products of these various methods exhibit differences, especially in their iodine values.

F. H. L.

Estimation of Carvone in Essential Oils. J. Walther. (*Pharm. C. H.*, 1900, xli., 613; through *Chem. Zeit. Rep.*, 1900, 313.)—Carvone is the valuable ingredient of the oils of mint and caraway. It may be determined by converting the ketone into oxime, and titrating the excess of hydroxylamine employed. Two to 5 grammes of carvone, or oil containing it, are mixed with 10 grammes of a fresh aqueous solution (2 : 3) of hydroxylamine hydrochloride; 25 c.c. of alcohol free from aldehyde and 2 grammes of sodium bicarbonate are added, and the whole is placed on a boiling water-bath under an upright condenser for half an hour. When cold, 6 c.c. of 1·12 hydrochloric acid are introduced, and the liquid is brought into a 500 c.c. flask, rinsing the apparatus with dilute HCl. The solution is diluted to the mark, filtered, and in 25 or 50 c.c. of the filtrate the unused hydroxylamine is titrated with decinormal sodium hydroxide, 1 c.c. of the latter being equivalent to 0·015 gramme of carvone.

F. H. L.

Investigation of the Value of the Halphen Colour Test for the Detection of Cotton-seed Oil. Rozier D. Oilar. (*Amer. Chem. Journ.*, xxiv., 355.)—The author has thoroughly investigated the reliability of the test. As recommended by him, it consists in heating 10 c.c. of the oil or melted fat under examination with 10 c.c. of amyl alcohol, and 10 c.c. of carbon disulphide containing 1 to 2 per cent. of dissolved sulphur in a boiling-tube, at first, with frequent agitation, in a steam-bath, and then, after the violent boiling has ceased, in a boiling salt-water bath (temp. 105 to 110°) for from forty-five minutes to three hours, according to the quantity of oil present, the tube being occasionally removed and shaken. As little as 1 per cent. of cotton-seed oil will give a beautiful crimson wine coloration in twenty minutes. If the mixture is heated for too long a time, a misleading brownish-red colour, due to burning, may be produced.

The reaction does not appear to be produced by any other of the common edible oils or fats, nor by the various fillers and colourings used with hog fats. It is more sensitive with fresh than with old fats, and a faint colour is still given by fresh lards containing only $\frac{1}{16}$ per cent. of cotton-seed oil, noticeable when compared with a blank test; with $\frac{1}{4}$ per cent. it is perfectly reliable.

The test may be directly applied to the soap or fatty acids of a fat mixture of high percentages of cotton oil, if not too deeply coloured, but the reaction is not so delicate. It may also be used as a test for free sulphur. The author gives tables showing the results obtained with various mixtures.

A. G. L.

Acid and Saponification Values of some Copals. W. Lippert and H. Reissiger. (*Zeits. angew. Chem.*, 1900, 1047.)—To determine the acid value, 10 c.c. of chloroform are poured upon 1 gramme of the copal, and after standing son hours 25 c.c. of alcohol are added. If the copal does not dissolve, it is necessary use ether, or ether and alcohol. When solution is complete, it is titrated with semi-normal alcoholic potash, using phenolphthalein as indicator.

To determine the saponification value, the copal is heated with alcohol and ether (chloroform must not be used), excess of standard alcoholic potash is added, and the determination is carried out as usual.

The ether value is the difference between the acid and saponification values. In the case of Manila copal, and perhaps in other cases, it is due, not to esters, but probably to lactonic bodies.

Copal.	Acid Value.	Saponification Value.	Ether Value.	Remarks.
Angola, white ...	59–61	—	—	Williams found acid value 57
„ red ...	90	148–152	58–62	Schmidt and Erban found saponification value 148
Angostura ...	48	—	—	Fine glassy yellow lumps
Benguela, elect ...	61–62	111–126	50–64	Clear yellow balls
„ ordinary	84–87	—	—	Contains dirt
Brazil, elect ...	153–158	148–157	1–5	Splendid pale yellow balls
Cameroon ...	106–109	—	—	Easily soluble in alcohol
Cowry, elect ...	81	—	—	Not quite transparent
„ light ordinary	63–64	95–109	32–39	Williams found saponification value 64·2
„ dark ...	66·5–70			
Dammar	33–35	—	—	Completely soluble in turpentine
Madagascar ...	60	—	—	Easily soluble in alc.-chloroform
Manila, elect ...	103–111	147–152	44–50	Hard, easily soluble in alcohol
„ sol. spirit	144–148	—	—	Soft, „ „ „
Sierra Leone ...	78–82	—	—	Small light balls
Zanzibar	—	88–102	—	Characteristic "goose-skin" appearance

Copals are practically always melted before being used for making lacquers, etc. The behaviour on melting is therefore the most important property to determine. The quantitative reactions described are, however, useful for detecting adulterants.

Artificial resins are made by heating colophony with glycerin, thus producing esters of the resin acids. The products thus obtained should have an acid value not higher than 20.

A. M.

Volumetric Method for the Estimation of Carbon Monoxide in Coal Gas. A. Smits, H. Raken, and P. C. E. M. Terwogt. (*Zeits. angew. Chem.*, 1900, 1002.) —After other constituents have been absorbed by potash, fuming sulphuric acid and phosphorus, the carbon monoxide is absorbed by means of the arrangement described by the authors. This consists of a U-tube filled with solid iodic anhydride. This tube is connected on the one side to the Hempel burette, and on the other to a pipette containing potash solution. The gas is passed several times backwards and forwards through the U-tube, by which means the carbon monoxide is oxidized to carbon dioxide, which is absorbed by the potash—$5CO + I_2O_5 = 5CO_2 + I_2$.

A. M.

INORGANIC ANALYSIS.

On the Qualitative Separation of Nickel from Cobalt by the Action of Ammonium Hydroxide on the Ferricyanides. Philip E. Browning and John B. Hartwell. (*Zeit. Anorg. Chem.*, 1900, xxv. 323.)—The authors urge two objections against F. W. Clarke's method (*Amer. Journ. Sci.*, xlviii., 67): first, the difficulty of obtaining a good filtration from the cobalt ferricyanide; and, second, the large amount of sulphur thrown down on the addition of ammonium sulphide to the filtrate.

The following modification does not suffer from these defects: To not more than 0·1 gramme of the mixed salts dissolved in 5 c.c. of water, a few drops of a solution of alum are added; the solution is then neutralized with ammonia, acidulated with acetic acid, 0·5 gramme of potassium ferricyanide added, the liquid agitated, and filtered after the addition of 5 c.c. of strong ammonia. Cobalt is recognised by the colour of the precipitated ferricyanide, and nickel is indicated by the black precipitate of nickelic hydroxide obtained in the filtrate on heating it to boiling with a small piece of sodium hydroxide. By this method, as little as 0·0002 gramme nickel can be detected in the presence of 0·05 gramme cobalt sulphate. A. G. L.

The Iodometric Estimation of Arsenic Acid. F. A. Gooch and J. C. Morris. (*Zeit. anorg. Chem.*, 1900, xxv., 227.)—By the action of a soluble arsenate on a soluble iodide in acid solution, the arsenic acid is reduced to arsenious acid more or less completely according to the conditions. In order to make the reaction complete, it is necessary to remove the iodine set free. This may be done either by running in thiosulphate or by evaporating down, the last traces being then removed by careful addition of sulphurous acid. After reduction the arsenic can be determined by neutralizing with a bicarbonate and titrating with iodine, the arsenious acid being thus reoxidized to arsenic acid.

Methods based on reduction with thiosulphate give inaccurate results, on account of other reactions taking place, whether the determination be made by direct titration with thiosulphate or by titration with iodine after neutralization. The iodide becomes oxidized by the air, and the thiosulphate is acted upon by the acid. On the other hand, by evaporating the iodine off accurate results may be obtained.

The arsenate is heated in an Erlenmeyer flask, with an excess of potassium iodide (0·5 gramme more than is theoretically required) and 10 c.c. of dilute sulphuric acid (1:1), the total volume of the liquid being 50 to 75 c.c. The liquid is boiled until iodine vapour can no longer be seen, and is then decolorized by the careful addition of sulphurous acid. It is now rapidly diluted with cold water and cooled, nearly neutralized with caustic potash, and then entirely neutralized with potassium bicarbonate. Finally starch is added as indicator, and the reduced acid is titrated with iodine solution. A. M.

Investigation of Millon's Reagent. W. Vaubel. (*Zeit. angew. Chem.*, 1900, 1125.)—This reagent is prepared by dissolving 1 part of mercury in 1 part of cold fuming nitric acid (specific gravity 1·4), warming and adding 2 parts of distilled water. With phenol and certain substituted phenols in aqueous solution it gives a red precipitate. The author finds that the reaction only takes place if there be unsubstituted hydrogen atoms in both an ortho- and a meta- position relative to the hydroxyl group. It does not take place, therefore, with di-ortho- or di-meta- substituted phenols. Of the naphthols and those of their derivatives examined, β-naphthol was the only one which gave the characteristic precipitate.

The reaction is due to the presence of mercurous nitrate and nitrous oxide. If sodium hydroxide be added, the red precipitate dissolves, giving a red solution.

A. M.

The Estimation of Tungsten in Steel and Steel-making Alloys. Fred Ibbotson and Harry Brearley. (*Chem. News*, lxxxii., 224.)—The powdered steel is heated with hydrochloric acid (100 c.c. for 5 grammes steel) just short of boiling, and a minimum amount of nitric acid gradually dropped in, until the sample has completely dissolved and all the iron is in the ferric condition. The solution is then boiled till WO_3 just begins to separate, diluted with twice its volume of water, and again boiled, when all the WO_3 except 2 or 3 milligrammes will be precipitated. In the ignited precipitate SiO_2 is estimated by means of HF, and then traces of F_2O_3 and Cr_2O_3, after fusion with sodium carbonate. The process is rapid, and results obtained with it are only about 0·05 per cent. too low.

Ferro-Tungsten Alloys.—As at most only 2 grammes can be handled, the error of the above method amounts to about 1 per cent. of the tungsten. For an accurate analysis the solution of the alloy is evaporated to dryness on the water-bath, the residue boiled with dilute hydrochloric acid, and the $WO_3 + SiO_2$ filtered off. Any molybdenum present will be in solution.

Nickel-Tungsten Alloys.—The powdered alloy is dissolved in HF and HNO_3, and evaporated with sulphuric acid till fumes of the latter come off. After diluting, WO_3 is filtered off, and nickel estimated by the cyanide process in the filtrate. For a complete analysis the sample is treated like a ferro-tungsten, molybdenum estimated as $PbMoO_4$, and nickel with potassium cyanide.

Tungsten-Molybdenum Steels.—The following method gives good results: The steel is dissolved as in the case of tungsten steels, the solution just evaporated to dryness, the residue boiled with dilute hydrochloric acid, WO_3 filtered off, and in the filtrate molybdenum and iron are separated with sodium hydroxide (*Chem. News*, lxxxi., 269).

Tungsten Powders.—The authors have noticed the presence of peculiar red crystals, possibly of tungsten bronze, which they have not been able to estimate accurately.

A. G. L.

Determination of Ferrous Iron in Minerals. W. F. Hillebrand and H. N. Stokes. (*Journ. Amer. Chem. Soc.*, 1900, 625.)—Mitscherlich's method of heating the mineral with sulphuric acid in a sealed tube gives high results, if any sulphides be present. This is due to the fact that the ferric iron is reduced by the sulphides. Accurate results are obtained by Cooke's hydrofluoric acid method. The silicate is decomposed by heating for an hour in a large platinum crucible on the water-bath, with dilute sulphuric and hydrofluoric acids. The cooled contents are poured into a platinum dish containing water, and rapidly titrated with permanganate nearly to the end. If sulphides be present, the solution is now filtered through a filter-paper supported on a platinum cone. The titration is then carried to the end. The pink colour will only be transitory, because the hydrofluoric acid acts on the excess of permanganate. A. M.

Estimation of Pyrrhotite in Pyrites Ore. F. B. Carpenter. (*Journ. Amer. Chem. Soc.*, 1900, 634.)—The presence of pyrrhotite (Fe_7S_8) is shown by its adhering to a magnet. The author's method for estimating it is based on the fact that pyrites (FeS_2) is practically insoluble in concentrated hydrochloric acid, whereas pyrrhotite is soluble. The quantity of material insoluble in hot concentrated hydrochloric acid is determined; from this the silica is deducted. From the percentage of pyrites thus obtained, and the total sulphur, the percentage of pyrrhotite is then calculated.

A. M.

A New Method for the Determination of Aluminium. E. T. Allen and V. H. Gottschalk. (*Amer. Chem. Journ.*, xxiv., 292.)—The method depends on the precipitation by carbon dioxide from the solution of an aluminate of a basic carbonate of aluminium containing a little alkali, which is converted into the normal hydroxide when boiled with water containing some ammonium salt. The authors state that the hydroxide obtained in this way is much denser and more easily washed than the ordinary precipitate obtained with ammonia, and that consequently much larger quantities can be handled. Accurate results are also obtained in the presence of sulphates, but alkaline earths and lithium must be absent.

For the determination the aluminium solution is nearly neutralized with ammonia, if acid, and a fresh solution of potassium hydrate run in from a burette until the precipitate is completely redissolved. The quantity of potassium hydrate added should be known, so that the amount of the impurities (silica, iron, alumina) may be deducted from the weight of alumina obtained. Through the alkaline solution a stream of carbon dioxide is then passed for some time, the precipitate transferred to a filter, washed, replaced in the original beaker, and boiled with about 200 c.c. of water containing a little ammonium chloride or nitrate for two or three minutes. The supernatant liquid is then decanted through another filter, using the filter-pump, the boiling and decantation repeated once or twice, and the aluminium hydroxide finally washed with water, ignited, weighed, re-ignited and reweighed. The average error of the method is less than 0·01 per cent. (0·045 to 0·004 per cent.).

A. G. L.

Determination of Free Alkali in Soap. R. E. Divine. (*Journ. Amer. Chem. Soc.*, 1900, 693.)

Total Free Alkali.—Two grammes of the sample are boiled for half an hour with 50 c.c. of alcohol and a measured excess of a standard alcoholic solution of stearic acid. The excess of acid is then determined by titration with caustic soda, using phenolphthalein as indicator. The acid absorbed is equivalent to the total free alkali.

Caustic Alkali.—Two grammes of the sample are dissolved in 50 c.c. of alcohol, a slight excess of barium chloride is added, and the solution is heated for a few minutes. It is then titrated with standard stearic acid solution, using phenolphthalein as indicator. The titration must be performed slowly with constant agitation. The acid added is directly equivalent to the caustic alkali. A. M.

A New Indicator for Acidimetry, and its Application to the Determination of Alkali-metal Carbonates and Boric Acid. J. Wolff. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1900, iii., 600-605.)—The author's proposal is based upon the fact that if salicylic acid be dissolved in an excess of sodium hydroxide with the addition of a little ferric chloride, and the excess of alkali be then titrated with sulphuric acid, a blood-red coloration changing to violet is produced when the point of saturation is reached. Observations show that the exact point of neutralization coincides with the production of the blood-red colour. The reaction is not only exceedingly delicate, but its sensitiveness increases with the dilution of the solutions. The author prepares the indicator as follows: Six grammes of sodium salicylate are dissolved in 25 c.c. of distilled water, and a dilute solution of ferric chloride is added drop by drop until a slight permanent turbidity is produced. The solution is then filtered, made up to 200 c.c., and divided into two equal parts, one of which is adjusted with soda to the point indicated by the production of a dark orange colour, and the other with acid to the point indicated by the production of a red. The two solutions are then mixed together, and 10 grammes of sodium salicylate added.

The author specially recommends the indicator for use in determining potassium and sodium carbonates. It at first colours solutions of these salts yellow, but as carbonic acid becomes liberated, the colour changes to red, and finally, with the slightest excess of sulphuric acid, to violet. He also applies it to the determination of boric acid in borax, and to the analysis of mixtures of boric acid and borax. To an exact quantity of the substance dissolved in water, a known excess of normal sulphuric acid is added, and the solution titrated with normal soda by the aid of the indicator. Glycerin is then added, and the solution again titrated with soda, using phenolphthalein as the indicator. The test results given are good. H. H. B. S.

REVIEWS.

Victor von Richter's Organic Chemistry; or, The Chemistry of the Carbon Compounds. Edited by Professor R. Anschutz, University of Bonn (assisted by Dr. G. Schrötter). Authorized translation by Professor Edgar F. Smith, University of Pennsylvania. Third American, from the eighth German, Edition. Volume II.: Carbocyclic and Heterocyclic Series. Pp. 671. London: Kegan Paul, Trench, Trübner and Co., Ltd., 1900. Price 15s.

The volume before us completes the third American edition of "Richter's Organic Chemistry." It deals not merely with aromatic compounds, *i.e.*, derivatives of benzene and allied substances, but with all compounds whose constitutional formulæ are represented by closed chains or rings. It may be mentioned, however, that the so-called alicyclic compounds (for example, certain lactones), which are closely related to, and derived by simple reactions from, members of the aliphatic series, are simply catalogued, their general properties having been dealt with in the first volume.

Bearing in mind that the literature of the aromatic compounds alone is more voluminous than that of all other departments of chemistry taken together, the scope of a work such as the one before us presents considerable difficulties. The editor certainly deserves the highest praise for the discretion he has exercised in this direction. Not only has the number of pages in the two volumes been kept remarkably even, but the matter is dealt with in a manner as it should be in companion volumes. We have already observed, in dealing with the first volume, that the work has, in a sense, outgrown the scope of a text-book, and it will doubtless serve many as a book of reference. In this connection the references to the literature form an important feature, and it is to be regretted that some of these refer to abstracts, and not to the original papers. Instances may be cited in the references given to G. Goldschmidt's work on papaverine, and that of W. H. Perkin junior on berberine.

The volume appears commendably free from typographical errors, and the translator has, on the whole, executed his task satisfactorily; yet the diction is not faultless, and we could point to several passages which are by no means clear.

A. R. L.

Chemistry: an Exact Mechanical Philosophy. By F. G. Edwards. Published by J. and A. Churchill, London. Pp. xii and 100. Price 3s. 6d.

By a space representation of the forms of atoms, the author seeks to explain their properties. He assumes that all matter is built up of tetrahedra: ether is supposed to consist of single tetrahedra, hydrogen of two tetrahedra joined together, and the other elements of a number of tetrahedra approximately twice the number expressing their atomic weight.

The mode of grouping the tetrahedra adopted gives a more or less well-marked periodic recurrence of similar forms with sunk or raised facets; the sunk facets predominating in the acidic elements, and the raised facets in the metallic atoms. The theory of molecules is rejected, and compounds are supposed to consist of atomic groupings, in which the metallic atoms with their raised facets vibrate to and fro between the sunk facets of the acidic atoms.

The theory is attractive, and will doubtless be accepted as the one and only solution of the laws of Nature by those who are able to reconcile the few seemingly adverse facts with the author's views. It is not strikingly obvious how the single tetrahedra composing the ether can be imponderable, despite the author's explanation, while the tetrahedra when grouped are ponderable. Many people will refuse to admit that the atomic weights of oxygen, chlorine, iodine and cadmium, generally accepted, are incorrect to about 1 per cent. of their value; others may fail to see any reason for the distortion of the tetrahedra, which occurs in all elements except hydrogen; and the sceptical may even ask whether the periodicity is not the result of choice of groupings rather than a natural sequence.

The work undoubtedly opens up a new vista in the domain of science, and the reader who masters the author's theory will not only broaden his mind, but will appreciate the great labour involved in the elaboration of the conclusions.

H. D. R.

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THE ANALYST.

FEBRUARY, 1901.

OBITUARY NOTICE.

THE LATE DR. W. SEDGWICK SAUNDERS.

On January 18 occurred the death of Dr. W. Sedgwick Saunders, who, since 1874, held the important positions of Medical Officer of Health and Public Analyst for the City of London, succeeding Dr. Letheby.

Dr. Saunders was born near Tavistock in 1824; studied medicine at St. Thomas's Hospital; entered the Army as Surgeon, and served in the West Indies and North America; then acted as Medical Officer to the Military Prison at Rochester. Afterwards he commenced practice as a surgeon in the City of London, and was elected to the Court of Common Council, on which he did good service until 1874.

As Medical Officer of Health he was instrumental in effecting numerous improvements in the sanitary condition of the City.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

NOTES ON CERTAIN BRITISH PHARMACOPŒIA TESTS.

By C. G. Moor, M.A., F.I.C., and Martin Priest, A.I.C.

(Read at the Meeting, November 7, 1900.)

We have recently been engaged in collecting data on the composition of commercial drugs and preparations commonly used in medicine, principally with the view of ascertaining how far they conform to the requirements of the British Pharmacopœia, 1898. The principal object we had in view was the collection of such information as would enable us to draw up suggested standards of purity, and tests which, taken in conjunction with those given in the British Pharmacopœia, would enable one to judge correctly as to the purity or otherwise of commercial drugs, for the purposes of the Food and Drugs Acts.

In studying the British Pharmacopœia from an analytical point of view, we have met with certain points which we think may be interesting to our fellow-analysts.

In two cases we find that the ash limits of the Pharmacopœia require revision, as, if the limits given were adhered to, they would result in the condemnation of genuine samples.

CARDAMOM SEEDS.

Thus, the British Pharmacopœia, 1898, gives a limit of ash of cardamom seeds of 4 per cent. This is intended to exclude mineral matter (small stones) in the unground fruits or the presence of powdered pericarps in powdered cardamom seeds.

We have, however, found that the genuine seeds removed from their pericarps by ourselves may give a higher ash than 4 per cent.

The following table shows the results obtained on various samples of cardamoms:

Ash on Whole Fruit.	Ash on Seeds.	Ash on Pericarps.
5·3	4·03	10·04
5·6	5·9	11·9
—	4·5	10·4
8·2	5·7	8·8
5·4	3·7	8·8
7·7	5·1	10·5
—	3·3	7·1
—	5·1	13·5
6·2	4·2	8·1
—	4·3	—
—	4·5	10·4

In order to distinguish between the powdered seeds and the powdered whole fruit, Greenish recommends a microscopic examination, and we think this might be supplemented by a fibre estimation. We have only determined these factors on one sample, and therefore no attempt must be made to generalize from the following figures:

Seeds.	Husk.	Whole Fruit.
11·0	47·2	21·2

In ashing the seeds and husks, it was noticed by W. Gladwyn that the ash of the husks differed from that of the seeds in being fusible, so estimations of soluble ash and its alkalinity were made, with the following results:

	Seeds.	Husks.
Total ash	5·0	13·3
Soluble ash	2·0	7·1
Soluble ash as K_2O	0·52	2·6

The ether extract was also determined on the whole fruits, and on the seeds and pericarps separately, but the results do not appear to be of any value as a means of distinction. In any further work on the subject, it should be remembered that the cardamoms of commerce vary much in quality, and both wild and cultivated varieties are sold, and that more than one species is in use. The subject is not one of great importance, as cardamoms are employed not so much for any medicinal value that they may contain, but as flavouring agents.

COLOCYNTH.

The colocynth apples of commerce consist of the peeled dried fruit of the *Citrullus colocynthis.* The fruit is imported chiefly from Smyrna, Trieste, France, and Spain. That from Spain is most used. The only part of the fruit that is official is the dried pulp free from seeds. The separation of the seeds from the pulp may be effected by proper means without much difficulty; but, according to Squire, commercially it is carried out very imperfectly, and he states that samples of the pulp commonly contain 10 per cent. of seeds.

The Pharmacopœia tests are as follows: "It should not yield the characteristic reaction with the tests for starch, and only traces of fixed oil should be removed from it by ether." It yields, when dried at 212° F. and incinerated, at least 9 per cent. of ash (indicating absence of seeds).

Squire (*Companion*) states that the seeds contain 15 per cent. of oil, which is in accordance with our own results. We have found that not merely oil, but also some gummy matter, is extracted by ether, both from the seeds and the pulp; hence it might be well to try the effect of a different solvent. We have obtained the following figures for the ash and the ether extract on colocynth pulp and seeds:

Ash on Seeds.	Ash on Pulp.	Ether Extract on Seeds.	Ether Extract on Pulp.
1·7	7·8	11·5	7·8
2·6	12·1	—	5·8
2·27	—	—	—
2·4	8·2	15·3	12·5
3·2	9·2	12·8	5·29
—	—	12·2	1·4
2·4	9·2	14·0	11·1

Additional estimations on pulp alone gave the following figures: 7·3, 8·6, 10·1, 7·2, 8·6, 7·8, 7·0, 10·5.

In all the above cases we carefully separated the seeds from the pulp in the laboratory. Before ashing the pulp, it is of importance to insure its complete desiccation at 100° C., as it is very hygroscopic. It is by no means an easy matter to insure complete incineration, and it would be far better that any future limit of ash should be specified to be the sulphated ash. All the ashes given in the table above were not sulphated, but were completely incinerated in a platinum dish.

It will be noted that from the Pharmacopœia description the pulp alone is official, but the powdered whole apple is sold as a purgative, and is said to be a favourite remedy amongst soldiers. The whole fruit powder is also sold as a moth powder.

With regard to other British Pharmacopœia tests, we venture to offer the following remarks:

OIL OF NUTMEG.

The Pharmacopœia directs, under Oleum Myristicæ, that "a little evaporated on a water-bath should not leave a residue which crystallizes on cooling" (absence of the concrete oil of nutmeg). At the beginning of the monograph, the Pharmacopœia defines Oleum Myristicæ as "the oil distilled from nutmeg." Strictly speaking, the oil which satisfies the Pharmacopœia is not the oil distilled from nutmegs, but the oil which has been fractionated so as to exclude the heavier portion. In a case of a sample of nutmeg oil being examined under the Act and proved to yield a crystalline residue, such an oil might rightly be considered as not in accordance with the Pharmacopœia requirements, but it could not be said that it was not genuine nutmeg oil.

PEPPERMINT OIL.

A somewhat similar case is that of peppermint oil. Among the tests given by the Pharmacopœia is the following: "If a portion of the oil be cooled to $-8{\cdot}3^\circ$ C., and a few crystals of menthol be added, a considerable separation of menthol should take place." Some perfectly genuine samples of oil fail to yield any deposit of menthol when treated in this manner, even though they may contain a considerable percentage of menthol, which may be estimated by acetylization. Such an oil is clearly not in accordance with the British Pharmacopœia requirements, but may nevertheless be genuine, and should not be condemned without consideration.

CASTOR OIL. CROTON OIL.

The colour test given in the British Pharmacopœia, namely, the behaviour of castor oil to sulphuric acid, requires revision. It states that the mixture should not become brown; but genuine oils do become brown, but not blackish-brown. Again, in croton oil, "the fresh oil" does not dissolve in alcohol as is required by the British Pharmacopœia, but after the oil has been kept for some time it is soluble, forming a clear solution.

BENZOIN. MYRRH. GALBANUM.

Certain other British Pharmacopœia descriptions or tests in the case of other drugs either require amendment or more careful definition; thus, benzoin is described as "almost entirely soluble in 90 per cent. alcohol." The words "almost entirely" should be replaced by a definite percentage. The following figures show the very varying amounts of matter insoluble in alcohol found in specimens of fair commercial quality, such as are used in the manufacture of tinctures, etc.

Source.	Amount insoluble in Warm 90 per Cent. Alcohol.
Sumatra ...	8·6, 15·6, 6·8, 6·5, 13·3, 18·1, 8·4, 10·8, 18·1, 8·4, 7·6

Three qualities of Sumatra gave the following results:

No. 1	9·2
No. 2	15·2
No. 3	16·7

In addition, a requirement as to the amount of benzoic and cinnamic acids in benzoin might reasonably be included. Myrrb, again, is a drug in which there is naturally a variable quantity of bark and woody matter; in this case the Pharmacopœia gives no test whatever, with the exception of a colour test to detect the presence of false myrrh. A reasonable limit of ash is 7 per cent., and of matter insoluble in alcohol 70 per cent. Gum galbanum often contains woody matter, and one sample recently examined by us contained as much as 46 per cent. of foreign matter.

Ammoniacum.

Ammoniacum is also liable to contain mineral matters, but no ash limit is given in the Pharmacopœia, and a limit for the matter insoluble in alcohol should also be included. In a previous paper we have dealt with the question of asafœtida, and showed that the greater portion of this drug now in commerce contains excessive quantities of mineral matter.

Strophanthus Seed.

The official strophanthus seed of the Pharmacopœia is the *Strophanthus Kombé*, and the only test given for it besides the description is that "sulphuric acid colours the endosperm, and sometimes the cotyledon, dark green." The Pharmacopœia ascribes the dark green colour to the presence of strophanthin. One might from this expect that a similar dark green colour would be obtained on treating the total solids of an evaporated tincture of strophanthus. We have examined sixteen samples of strophanthus tincture, and in all of these a certain amount of dark green colour was produced; in many cases, however, a red colour predominated, and as there are non-official strophanthus seeds which give a red colour with sulphuric acid, one might conclude that tinctures yielding a red colour with sulphuric acid were prepared from a non-official seed. We therefore obtained specimens of undoubted *Strophanthus Kombé* and prepared tinctures ourselves; these also yielded a mixture of red and green colours when the dried residue was streaked with sulphuric acid.

In no case was the colour entirely green; these results are confirmed by those of Perédès (*Pharm. Journ.*, August 25, 1900, p. 241), who found that the official seed may yield a red colour as well as a green when tested with sulphuric acid.

It must not, therefore, be concluded that a tincture yielding streaks of red as well as green has not been prepared entirely from the official seed.

Strophanthus Tinctures.

Total Solids. Per Cent.	Specific Gravity.	Colour Test.
0·33	0·894	Red and green.
0·42	0·889	Red and green.
(1885) 0·70	0·835	More red than green.
0·41	0·890	Nearly all green.
0·47	0·890	Red, some green.
0·45	0·889	Red.
0·59	0·890	Nearly all green.
(1885) 0·65	0·837	Green and red.
0·46	0·889	Mostly red, some green.
0·52	0·891	Chiefly green, with some red.
0·42	0·896	Red, some green.
0·74	0·894	Red, little green.
0·21	0·883	Chiefly green, some red.
0·48	0·893	Red, some green.
0·40	0·893	Red and green.
0·68	0·891	——
0·44	0·893	——

In the case of several drugs ash limits might with advantage be fixed. We have also recently drawn attention (Pharmaceutical Conference, July, 1900) to the excessive amounts of mineral matter, due to imperfect cleaning, which are often found in arnica, serpentary and valerian roots.

ARNICA ROOT.

In the case of arnica root, clean specimens free from dirt give about 8 per cent. ash, but commercial samples often yield as much as 25 to 30 per cent., owing to the earth mechanically enclosed by the rootlets.

SERPENTARY ROOT.

Clean serpentary root gives an ash of about 8 per cent., whereas one specimen gave an ash of 30 per cent., and two samples purchased in powder gave 18 per cent. ash each.

VALERIAN ROOT.

Valerian root when clean also yields about 8 per cent. ash, but in three commercial samples we found 13, 15 and 19 per cent., while a specimen of the powdered root yielded 29 per cent.

ARAROBA.

Araroba is well known as a drug in which the mineral matter is very variable, and no ash limit is specified in the British Pharmacopœia; if, however, it satisfies the Pharmacopœial test of yielding to chloroform not less than 50 per cent. of chrysarobin, the sample may be regarded as of good character.

PYRETHRUM, STAVESACRE, COCCUS CACTI.

In addition to these, other drugs, particularly those in powder, occur in commerce with excessive amounts of mineral matter. As examples of these, we may cite pyrethrum root, two samples containing 18 and 19 per cent. of ash, as against the 5 or 6 per cent. which is natural; stavesacre seeds, containing 26 per cent., whereas the normal is about 12 per cent.; *Coccus cacti*, containing 30 and 41 per cent. of ash, whereas the Pharmacopœia limit is 6 per cent., and the true ash under 4 per cent., the high mineral matter in these cases being intentional additions of French chalk. The attention of analysts may be well directed to this article, for, whether sold for the purpose of medicine or not, the existence of so gross a sophistication is a disgrace.

SAFFRON.

Saffron has been adulterated for centuries, both with mineral and vegetable matters. The tests given in the British Pharmacopœia are satisfactory so far as they go, but should be supplemented by the following: "Every fragment should on contact with strong sulphuric acid afford a deep though transient blue colour." The dyed fragments which we have found gave a pink colour to sulphuric acid instead of the blue afforded by true saffron.

SANTAL-WOOD OIL.

Capsules are sold as containing santal-wood oil which really contain cedar-wood oil or West Indian santal oil, which is derived from a totally different tree to the true santal-wood, and possesses altogether different analytical characters.

Both these adulterants are readily distinguished from genuine santal oil by the ordinary tests and by the colour test with bromide of tin, published by one of us in the ANALYST of August, 1895, p. 174.

SCAMMONY.

Scammony is to be found at the present day adulterated with starch, and out of six samples recently examined only one was genuine.

TINCTURES.

In studying the tinctures of the Pharmacopœia, we have estimated the total solids on a large number of samples (several hundreds), many of which we know to have been prepared strictly in accordance with the official directions.

As the total solids would, in many cases, be taken as a criterion of the correct preparation of the article, particularly in those instances where there is no active principle that can be conveniently estimated, we would point out that, while the solids are remarkably constant in some tinctures, in others there are much greater variations than might be expected. This applies also to the liniments and the liquid extracts of the Pharmacopœia, so that no conclusion should be drawn from the amount of total solids yielded by such preparations, until a large number of figures have been accumulated.

NOTE ON THE ESTIMATION OF GLYCEROL.

By J. LEWKOWITSCH, PH.D.

(*Read at the Meeting, December* 5, 1900.)

A few years ago Laborde* published a method for the determination of glycerol in fermented liquors, which was based on the following reaction:

$$C_3H_8O_3 + H_2SO_4 = C_3 + SO_2 + 5H_2O.$$

The carbon was supposed to separate out quantitatively, and after suitable washing with hydrochloric acid and treatment with ammonia and heating until the ammonium salts were volatilized, the pure carbon was weighed and calculated to glycerol.

I suppose most of us have been shaking our heads over this method, and I confess that I did not consider it seriously until F. Jean† proclaimed it an important advance in the estimation of glycerol. Indeed, he recommends it for the determination of glycerin in soaps, and fats and oils generally.

I had occasion in my laboratory to examine this method, and the results obtained by following the directions given by Jean are set out in the following table:

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* *Ann. Chim. Analyt. Appliquée*, iv., 76, 110.

† *Ibid.*, v., 211.

Sample.	Glycerol found by Acetin Method.	Glycerol found by Bichromate Method.	Glycerol found by Carbon Method.	
			Including Ash.	Free from Ash.
	Per Cent.	Per Cent.	Per Cent.	Per Cent.
No. 1	—	13·79	4·76	
			13·61	
			12·76	
			16·26	
			10·96	
No. 2	86·21	—	89·54	88·39
			68·23	67·23
			73·2	72·25
			59·13	58·61
			77·73	77·2
			83·07	81·74
No. 3	80·47	—	68·9	64·95
			57·35	54·41
			85·60	82·07
			69·26	68·09
			97·98	88·72
			44·50	40·60
No. 4	81·31	—	76·92	75·9
			80·36	79·0
No. 5	—	—	3·39	3·28
			3·19	3·03
			4·1	3·98
			1·18	1·01
			0·29	0·18

I think a glance at the figures shows that you can obtain any result you please, and it is easy to regulate the percentage if one desires to do so, for it is almost impossible to prevent the finely divided carbon from firing, however careful one is in driving off the ammonium salts.

It is clear that the carbon will be more or less contaminated with ash, and in a number of experiments the ash has been deducted.

It is very unpleasant to have to publish figures of this kind, but if I can elicit more correct directions from the authors of this method, or, if that be impossible, if I can prevent some of my colleagues from spending as much time as my assistants have done, I think I may claim to have done them some little service.

My thanks are due to Messrs. C. D. Robertshaw and G. Warburton, who have carried out the determinations.

ON THE EXAMINATION OF VARNISH RESINS.

By J. Lewkowitsch, Ph.D.

(*Read at the Meeting, December 5, 1900.*)

I think it will be generally admitted that the analysis of varnishes is a *terra incognita*, if it is a question of determining by chemical means what gum has been used in the preparation of an oil varnish. In his despair the analyst will try to use the methods which have proved of such enormous advantage in fat analysis, and, as is well known, a number of determinations of the acid value, saponification value, and iodine values have been carried out, but their use for the diagnosis of gums in mixtures such as we have to deal with is almost infinitesimal.

It struck me that it has been generally* overlooked that the determination of the so-called "constants" has been carried out on the original gums as obtained in the market, whilst the gums are, of course, subjected to a process of "running" or melting, which is accompanied in some cases by destructive distillation.

In the course of an examination of varnishes in my laboratory I had occasion to collect a few numbers representing the equivalent of what is termed in fat analysis "acid value," "saponification value," etc., and I have placed side by side with the numbers obtained with the most frequently used resins in the original state, those found after heating them to 300° C.

The result is very discouraging indeed, and I should certainly not have published the figures if I had not been asked to do so by a scientific varnish-maker, who thinks that these figures will be of value. So far, I cannot see that they will help in the analysis of varnishes, and the present paper must, therefore, be looked upon as merely putting on record some numbers such as anyone might get by a cursory examination.

I may add that the "acid value" was determined in the same manner as is done in fat analysis, with finely powdered material, regardless of whether the gum dissolved completely or not. If an excess of aqueous caustic potash is added, and back titration resorted to after some time, higher numbers are obtained, and it is possible to arrive at any figure for the acid value, provided one allows to stand long enough, and keeps adding fresh excess of alkali after neutralization, and again allows to stand. In some cases acid values were thus obtained which were far higher than the saponification value, a fact which would point to the gradual degradation of the constituents of the gums. The arbitrary fixing of a certain time during which an excess of alkali is allowed to act on the gum would therefore appear to lack scientific foundation.

As the bromine addition method was done in most cases in acid solution, the bromine substitution value was not determined throughout. This will be done in future.

I think I have made it sufficiently clear that I do not attach much value to the figures given in the tables for purposes of detecting the gums used in the preparation of an oil varnish, quite apart from the fact that commercial samples show the greatest possible divergencies. Perhaps other tests I have in hand may lead to more useful information (such as oxygen absorption, Twitchell's resin determination, Liebermann's starch colour reaction, and a few more that readily suggest themselves to the analyst).

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* Compare, however, R. Williams, Analyst, 1898, 254.

I am indebted to my assistants, Messrs. C. D. Robertshaw and G. Warburton, for the figures in the following table:

	ORIGINAL GUMS.						GUMS AFTER HEATING TO 300° C.				
	Acid Value.	Sap. Value.	Unsap., %	Iodine Value.		Sol. in Alcohol, %	Acid Value.	Sap. Value.	Unsap., %	Iodine Value.	
				ICl.	Br.					ICl.	Br.
Copal, Commercial ...	109·8	143·1	7·96	135·5	183·6	93·06	26·49	85·4	14·73	124·3	181·3
Copal, Commercial ...	42·43	66·82	14·99	191·2	114·6	54·83	24·94	61·04	46·40	143·7	233·5
Copal, Sierra Leone ...	72·83	119·03	18·81	105·7	96·05	—	12·89	114·9	17·22	125·5	173·6
Copal, Manilla ...	127·6	175·17	15·98	137·9	188·2	—	68·21	136·3	22·99	133·3	186·4
Copal, Brazil	108·99	171·4	7·66	127·7	72·66	—	46·25	113·8	38·74	136·7	225·9
Copal, Sierra Leone ...	65·7	110·5	16·27	94·55	117·0	—	15·32	123·8	22·31	95·23	135·4
Cowrie ...	37·39	53·84	20·07	90·99	—	—	17·14	61·07	10·39	67·70	74·71
Mastic ...	52·73	81·79	51·13	175·7	185·0	84·41	23·23	50·24	49·28	165·0	217·8
Shellac, Dark	61·13	203·0	3·56	35·57	13·25						
Sandarac, Mogador...	134·39	143·42	13·2	112·2	99·98	—	64·84	136·14	14·28	126·4	69·13
Sandarac, Austral. ...	131·15	134·32	17·44	125·4	63·83	—	106·6	137·26	9·93		
Animis No. 1	18·69	73·15	6·3	105·3	182·7	—	9·25	58·73	—	106·1	207·4
Animis No. 2	30·22	93·5	6·85	96·21	95·85						
Dammar ...	35·22	32·73	75·96	127·5	169·4	—	10·85	60·44	86·57	127·0	196·8
Amber ..	16·7	121·27	18·86	58·98	82·82						
Succinit* ...	11·24	113·68	7·06	55·05	78·4	—	10·60	99·19	16·25	74·34	147·8
Colophony...	—	—	—	—	—	—	146·46	153·01	15·94	133·7	302·1

DISCUSSION.

Mr. HEHNER agreed with Dr. Lewkowitsch that it was useless to examine the original gums, as they, on boiling, underwent considerable chemical change. The difficulty was rendered greater by the fact that the gums, as a rule, were not heated by themselves, but were mixed with oil. It was interesting to notice that a gum which, like mastic, had as much as 51—or of 49 after heating—of unsaponifiable matter, nevertheless had an iodine absorption of 175 before heating, or 165 after heating. This seemed strongly to suggest that the unsaponifiable matter itself had a high iodine absorption.

Mr. A. MARSHALL said that, in the determination of bromine absorption, working with carbon tetrachloride in the manner which he had described in a recent paper before the Society of Chemical Industry (*Journ. S. C. I.*, March, 1900), he had lately obtained some very interesting results; and he thought that possibly a similar process applied to some of these gums might yield results of value.

Dr. RIDEAL suggested that more might be learnt as to the changes which took place in these gums as the result of heating by taking one particular sample of gum and tracing the changes produced by heating it to different temperatures.

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* This is a species of amber.

Mr. C. T. Tyrer said that there were on the market so-called varnish gums which were practically manganese resinates, containing considerable proportions of manganese; and probably in such cases the ordinary methods of analysis could not be successfully applied.

Mr. Mitchell inquired whether the author had tried amyl alcohol as a solvent in the determination of the saponification value. Its use was said to produce values differing considerably from those obtained with ordinary alcohol.

Mr. Marshall said that in determining the acid value it was necessary, as nearly as possible, to dissolve the gum entirely, and some solvent, or mixture of solvents, should be used by means of which it was possible to effect this.

Dr. Lewkowitsch said that where only one gum was present the matter was comparatively easy to deal with, but when the gum consisted of a mixture of various copals, for instance, the question of identifying them was almost impossible to solve; for even among gums which were sold by the same name, trade samples varied so much that experts, judging by external indications, were frequently misled. In addition, information was in many cases not obtainable as to the treatment to which samples had been subjected, owing to the secrecy which prevailed in the trade. He was afraid that an investigation of the unsaponifiable matter would not prove of very much assistance. The figures were more or less accidental, depending mainly on the time of boiling with caustic potash. In some cases the figures were widely divergent, though in others—in the case of sandarac especially—they were more concordant. He was afraid that the adoption of Mr. Marshall's suggestion would not result in any improvement. As a matter of fact, the choice of an acetic acid solution in the bromine absorption determination in these particular cases had been due merely to a personal dislike of the use of carbon disulphide or tetrachloride; but the whole matter had been thoroughly investigated, and in some cases bromine addition values and bromine substitution values had been determined, although they were not given in the tables. He had not been able to spare sufficient time to go further into the matter and employ a menstruum which could completely dissolve the gums, and the question of time also had led him to adhere to one temperature, namely, that which was most commonly adopted in operations conducted on a large scale. He ought, perhaps, not to have referred to "varnish gums," but to "gums used in varnish-making." All these gums were of course added to oils which were afterwards boiled with certain proportions of manganese resinate, or linolate, etc., and the metal was removed in the course of analysis. He had not tried amyl alcohol as a solvent, but he would be surprised if it were actually a fact that higher figures were obtained by its use, provided the saponification in alcoholic solution was carried out properly. The only explanation would seem to be that it was not pure.

THE COMPOSITION OF INDIAN COWS AND BUFFALOES' MILK.

BY J. WALTER LEATHER, PH.D., F.I.C.

INFORMATION as to the composition of Indian cows and buffaloes' milk will doubtless be of interest to members of the Society of Public Analysts, and I submit now the following results of analyses made by myself. I may mention than an idea had become current in India that the milk of Indian cows is different in composition to that of European cows, and accordingly complete analyses were in each case made. As to the composition of buffaloes' milk, no reliable information existed previously.

In Table I. are given analyses of milking from a number of cows at the Poona Dairy Farm (Bombay Presidency), and in Table II. similar information regarding milk from cows of the Saidapet herd (Madras). Table III. exhibits the composition of buffaloes' milk (Poona herd).

Firstly, as to Indian cows' milk. It is found to differ in no essential particulars from that met with in Europe. The relations existing between the specific gravity, the solids-not-fat, and the fat agree well in all cases with those which Richmond and others have found in the case of milks from English cows. So also, as shown in Table IV., the relationship between the milk-sugar, the proteids, and the mineral matter agrees well with the proportions 13 : 9 : 2. The percentage of butter-fat is high, varying from 4 to 6. Indian cows' milk is invariably very nearly white and the butter very pale yellow, unless coloured artificially.

Secondly, as to buffaloes' milk. It will be seen that the majority of the samples analysed are characterized by an extraordinarily high proportion of butter-fat, 7 and 8 per cent. being common, and in one case close on 10 per cent. of fat was found. Buffaloes' milk is white and the butter is also usually quite white.

As will be seen from Table III., the relation existing between the specific gravity, the solids-not-fat, and the fat is the same as for cows' milk, the solids-not-fat, when calculated by Richmond's formula, agreeing well with those found by analysis.

The percentage of proteids in buffaloes' milk is distinctly higher than in cows' milk, and varies from 3·5 to 4·3 per cent. The percentages of milk-sugar and mineral matter correspond very closely with those of cows' milk. On the other hand, the relationship between these three ingredients is not the same as for cows' milk. In one particular sample (that of the mixed milk of the herd) the percentage of proteids was higher than I found in most of the individual milks; but this does not materially alter the fact. If one takes the mean figures, the relationship works out to about 10 : 12 : 2. It is probable that the proportion of proteids is necessarily higher in a milk containing so much butter-fat, in order that the albuminoid ratio may be maintained.

TABLE I.—COMPOSITION OF INDIAN COWS' MILK. POONA HERD (BOMBAY), 1899.

	Janki, March 1.	Sind Cows.							Half-bred Deccani-Kalyani, 31/1/99.	Aden Cows.		Aden-Sind Yam-meni, 4/2/99.	Average Milk of Cows, 6/2/99.	Average Milk of Cows, 28/2/99.
		Janki, 24/1/99.	Sama-rath, 25/1/99.	Sama-rath, 26/1/99.	Sama-rath, 27/1/99.	Sunda, 29/1/99.	Sabin, 30/1/99.	Bhawa, 1/2/99.		Ner-mada, 2/2/99.	Kar-angi, 3/2/99.			
r...	87·35	87·51	85·76	85·88	85·67	86·82	84·72	85·27	84·89	86·68	87·27	86·02	86·35	86·32
...	3·46	3·65	4·89	4·88	5·20	4·10	6·11	5·59	5·77	4·43	4·28	4·64	4·75	4·91
ids	3·65	3·16	3·68	3·61	3·54	3·14	3·62	3·55	3·89	3·06	2·76	3·35	3·31	3·33
ar..	4·88	4·65	4·37	4·56	4·56	4·58	4·13	4·65	4·04	4·85	4·80	4·86	4·63	4·58
ral tter	0·72	0·68	0·71	0·78	0·86	0·69	0·77	0·67	0·74	0·69	0·66	0·83	0·70	0·73
	100·06	99·65	99·41	99·71	99·83	99·33	99·35	99·73	99·33	99·71	99·77	99·70	99·74	99·07
s- -fat	9·19	8·84	9·35	9·24	9·13	9·08	9·17	9·14	9·34	8·89	8·45	9·34	8·90	8·77

TABLE II.—COMPOSITION OF COWS' MILK. SAIDAPET HERD (MADRAS), 1900.

	Average Milk of Herd, 29/3/00.	Average Milk of Herd, 4/4/00.	Average Milk of Herd, 7/4/00.	Baggeon II., 28/3/00.	Govindi, 2/4/00.	Parvati, 3/4/00.	Manachu II., 5/4/00.	Poongan, 6/4/00.	Manachu III., 9/4/00.
er	86·27	86·87	85·97	86·14	85·52	84·48	86·13	86·98	86·08
... ...	4·00	4·14	4·68	4·41	5·48	6·07	4·57	3·52	4·62
teids	3·15	3·16	3·23	3·23	3·14	3·27	3·44	3·27	3·25
k-sugar ...	4·82	4·81	4·92	5·03	4·68	4·66	4·56	4·96	5·05
eral matter ...	0·76	0·74	0·72	0·76	0·70	0·72	0·77	0·75	0·77
	99·00	99·72	99·52	99·59	99·52	99·20	99·47	99·48	99·77
ids-not-fat ...	9·73	8·99	9·35	9·45	9·00	9·45	9·30	9·50	9·30
ecific gravity at 15·5° C.	1033·50	1032·0	1032·0	1033·0	1031·0	1033·0	1032·0	1034·0	1033·0
ids-not-fat calculated by Richmond's formula	9·32	8·97	9·08	9·27	8·99	9·60	9·05	9·34	9·31

TABLE III.—INDIAN BUFFALOES' MILK. POONA HERD (BOMBAY).

	Jaffarabadis.				Suratis.		
	Nevasi, 6/2/99.	Nevasi, 13/2/99.	Champa, 7/2/99.	Thami, 10/2/99.	Kevada, 8/2/99.	Phonga, 9/2/99.	Mangel, 11/2/99.
ater	86·43	85·09	82·84	84·05	82·63	83·38	81·74
t	4·08	5·78	7·32	6·89	7·74	6·86	8·66
oteids	3·29	3·24	4·36	3·53	3·75	3·99	4·32
ilk-sugar	5·12	5·09	4·42	4·70	4·94	4·70	4·66
ineral matter ...	0·80	0·68	0·75	0·82	0·68	0·72	0·80
	99·72	99·88	99·69	99·99	99·74	99·65	100·18
lids-not-fat ...	9·49	9·13	9·06	9·63	9·63	9·76	9·60

TABLE IV.

	Nevasi, 28/2/99.	Gaura, 2/3/99.	Jamadar, 3/3/99.	Kaoli, 3/3/99.	Bhadra, 4/3/99.	Asman, 4/3/99.	Gulab, 6/3/99.	Mixed Milk of Kirkee Buffaloes.	Rekhmi, 6/3/99.	Avadi, 7/3/99.
Water	85·78	82·16	80·16	81·57	82·89	81·15	82·63	82·22	82·74	82·52
Fat	5·17	8·14	9·95	8·79	7·56	8·81	7·59	8·09	7·42	7·52
Proteids ...	3·31	4·33	4·04	3·86	3·62	4·31	4·39	4·34	3·91	3·89
Milk-sugar ...	4·97	4·27	4·58	4·66	4·87	4·61	4·89	4·56	4·65	4·60
Mineral matter	0·70	0·77	0·79	0·71	0·71	0·76	0·73	0·76	0·81	0·80
	99·93	99·67	99·52	99·59	99·65	99·64	100·23	99·97	99·53	99·33
Solids-not-fat ...	9·05	9·70	9·89	9·64	9·55	10·04	9·78	9·69	9·84	9·96
Specific gravity at 15·5° C. ...	1034·5	1032·1	1031·2	1032·0	1032·9	1032·9	1032·7	1032·1	1034·3	1033·8
Solids-not-fat, calculated by Richmond's formula ...	9·79	9·79	9·93	9·90	9·87	10·12	9·83	9·78	10·19	10·09

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

The Detection of Artificial Red Colouring Matters in Wine. J. Bellier. (*Ann. de Chim. anal.*, 1900, v., 407-413.)—For the detection of archil, ammoniacal cochineal, phytolacca, or beetroot-juice in wine, the author advocates the use of a reagent consisting of: Mercuric oxide, 5 grammes; ammonium sulphate, 10 grammes; and ammonium hydroxide (specific gravity, 0·920), 15 c.c.; with sufficient water to make the solution up to 50 c.c.

In testing a wine, 10 c.c. are shaken with 1 c.c. of this reagent and filtered. The filtrate from pure wines is colourless, yellow, or sometimes gray, with a tinge of blue or green in the case of wines containing very little acid. Wines artificially coloured, on the other hand, yield a filtrate of a more or less pronounced red colour.

Instead of this reagent, the two following solutions may be employed: (*a*) Mercuric chloride, 10 grammes; ammonium chloride, 5 grammes; water, 100 c.c. (*b*) Glacial acetic acid, 10 c.c.; water, 65 c.c.; ammonium hydroxide (0·920 specific gravity), 25 c.c.

Ten c.c. of the wine are shaken with 1 c.c. of (*a*), then 1 c.c. of (*b*) added, and the whole shaken and filtered. The reaction is a little more sensitive than the first one, but requires greater care. The filtrate must be nearly neutral, and its colour must be noted immediately after filtration, since with some wines it becomes orange on standing.

Archil, when present, is partially retained by the precipitate in either test, but can be dissolved off the filter by absolute alcohol.

To determine to which of the four colouring matters the red colour is due, the filtrate, preferably as obtained by the second method, is divided into two parts. To one of these is added an excess of clear lime-water, whilst an excess of freshly-calcined magnesia is added to the second. The tubes are left for thirty minutes, with occasional agitation, after which an excess of acetic acid is added to the first tube.

1. If the liquid regains its red colour, archil or cochineal is present. To distinguish between them, 10 c.c. of the wine are mixed with 0·2 to 0·3 gramme of stannous chloride and an excess of finely-powdered calcium carbonate, shaken at intervals for ten to fifteen minutes, and filtered. If the filtrate is red, cochineal is indicated; if colourless, archil.

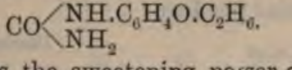
2. If the colour is not restored by acetic acid, phytolacca or red beetroot is indicated.

The filtrate of the tube containing the magnesia is mixed with acetic acid in excess and filtered, a colourless or yellow filtrate indicating phytolacca, whilst a restoration of the red colour points to the presence of beetroot. This reaction is not always successful.

As a further test, 10 c.c. of the wine are boiled with 1 c.c. of a reagent prepared by dissolving 40 grammes of powdered ammonium sulphate and 20 grammes of mercuric oxide in a little water, and making the solution up to 100 c.c. The filtrate from this test is colourless or yellow in the presence of phytolacca or beetroot, and red with archil or cochineal.

C. A. M.

The Detection and Estimation of Dulcine in Beverages. J. Bellier. (*Ann. de Chim. anal.*, 1900, v., 333-337.) Dulcine or sucrol is phenetol carbamide.



$$CO\left\langle\begin{array}{l}NH.C_6H_4O.C_2H_6.\\ NH_2\end{array}\right.$$

It possesses about 200 times the sweetening power of sugar. It is only slightly soluble in water (1·25 gramme per litre), ether, benzene, and chloroform, but dissolves readily in alcohol, ethyl acetate, and amyl acetate.

To extract it from lemonade, dilute fruit-syrups, etc., all that is necessary is to shake the liquid, after being made alkaline, with ethyl acetate. But in the case of beer, wine, and certain fruit-syrups, an emulsion is formed and other substances are also extracted. To obviate this, fruit-syrups, preserves, etc., are diluted, an excess of basic lead acetate added, the lead removed by precipitation with sodium sulphate, and the filtrate made alkaline, and extracted with ethyl acetate.

Wine is best treated by adding 2 grammes of mercuric acetate, shaking, adding a slight excess of ammonium hydroxide, and filtering.

In the case of beer, 2 to 3 grammes of pulverized sodium phosphotungstate and 10 to 20 drops of sulphuric acid are added to 200 c.c., the liquid allowed to stand for a few minutes after shaking, and then filtered, and the filtrate rendered alkaline with ammonium hydroxide.

The clear filtrate obtained by one of these methods is shaken with neutral ethyl acetate in the proportion of 50 to 200 c.c., the extract filtered, and the solvent evaporated. In most cases crystals are obtained when the quantity of dulcine exceeds 0·04 to 0·05 gramme per litre. If necessary, the residue is purified from fat and resinous substances by repeatedly treating it with hot water, filtering, and evaporating the solution to dryness. When sufficiently pure the final solution in hot water is evaporated to dryness in a porcelain basin, the residue treated with 1 to 2 c.c. of sulphuric acid and several drops of formaldehyde, and after fifteen minutes the mixture diluted with 5 c.c. of water. In the presence of dulcine a more or less pronounced turbidity or precipitate is obtained. When it only amounted to 0·01 gramme per litre the reaction is not very pronounced, and in such cases should be confirmed by Jorissen's reaction.

The purified dulcine is dissolved in 2 to 3 c.c. of boiling water, and the solution heated on the water-bath for five minutes with 4 or 5 drops of a solution of mercuric nitrate, prepared by dissolving 2 grammes of mercuric oxide in dilute nitric acid, adding sodium hydroxide solution until a slight permanent precipitate is formed, diluting to 15 c.c., and decanting the clear liquid. The tube is then withdrawn from the bath, no notice being taken of any precipitate, and a small quantity of lead peroxide introduced. The mercuric compound and the excess of the lead peroxide rapidly subside, and when dulcine is present to the extent of 5 milligrammes per litre the upper layer has a distinct violet colour.

For a quantitative estimation, any alcohol must be eliminated from the liquid, and two extractions with ethyl acetate made. The residues are united, purified, and dissolved in 1 to 5 c.c. of sulphuric acid, several drops of formaldehyde added, and the liquid diluted to 10 to 50 c.c. after 50 minutes.

After twenty-four hours the precipitate is collected on a dried filter of known weight, washed with water, dried and weighed.

By this process the author has obtained the following results with beverages containing 0·1 gramme of dulcine per litre:

200 c.c. of	Insoluble Compound. Gramme.
Lemonade	0·018
,, ...	0·017
Wine	0·020
Beer	0·021

Saccharin does not form an insoluble compound under these conditions.

C. A. M.

The Estimation of Gluten in Flour. Marion and Manget. (*Ann. de Chim. anal.*, 1900, v., 249-252.)—A method of estimating the value of a flour for baking purposes has been based upon a determination of the ratio of gluten to gliadin by Fleurent (*Bull. du Ministère de l'Agriculture*, December, 1899), and subsequently a similar process has been devised by H. A. Guess (Analyst, xxv., 267). Fleurent's method consists in kneading in the usual manner 33·33 grammes of the flour, and triturating the residual gluten with a small quantity of 70 per cent. alcohol containing 0·3 per cent. of potassium hydroxide. The mass is then transferred to a stoppered flask with the aid of the alkaline alcohol, the total amount of which should be 80 c.c. The mortar is rinsed with 70 per cent. alcohol (free from alkali), the washings being added to the contents of the flask, which are now shaken with glass beads until the disintegration is complete. After neutralizing the alkali with carbon dioxide, the liquid is made up to 150 c.c. with 70 per cent. alcohol, and filtered. Fifty c.c. of the filtrate are evaporated to dryness, and the residue dried at 100° to 105° C. and weighed. The amount of potassium carbonate present is deducted, the difference giving the amount of gliadin. The gluten is then obtained by calculation.

In the modification recommended by the authors, the gluten from the kneading is allowed to dry in the air and weighed. It is then shaken for two minutes in a glass vessel with 80 c.c. of Fleurent's solution and a dozen glass balls of 1 centimetre in diameter, and subsequently for fifteen to twenty minutes after the introduction of some small glass beads.

The turbid liquid is poured off into a flask with a long neck, graduated from 200 to 210 c.c. in tenths of a c.c., the flask and residual glass balls being washed with exactly 120 c.c. of pure 70 per cent. alcohol. The excess of the total liquid over 200 c.c. represents the volume of gluten. Fifty c.c. are evaporated, and the residue dried at 100° to 105° C., and weighed, giving, after making a deduction for the potassium hydroxide present, the weight of dry gluten. This is calculated on the volume read on the graduated flask. The residual liquid is neutralized with carbonic acid, and the gliadin estimated in 50 c.c. of the filtrate by Fleurent's method.

The following results are given in illustration :

	Hungarian Groats.		Superior Flours.				
			1.	2.	3.	4.	5.
Gluten, moist, per cent.	22·85	22·28	26·37	20·70	23·08	21·02	20·54
,, dry ,, ...	8·42	7·86	8·69	6·89	7·84	7·19	7·26
,, ,, by weight	8·39	7·56	8·93	6·79	7·67	6·91	7·47
Gliadin	5·52	5·23	5·57	4·79	4·50	4·63	4·97
Ratio : $\frac{\text{gluten}}{\text{gliadin}}$	$\frac{25}{48}$	$\frac{25}{56}$	$\frac{25}{41 \cdot 44}$	$\frac{25}{59 \cdot 35}$	$\frac{25}{35 \cdot 48}$	$\frac{25}{50 \cdot 76}$	$\frac{25}{49 \cdot 50}$

C. A. M.

ORGANIC ANALYSIS.

The Use of Tannin in purifying Residues containing Alkaloids. C. Kippenberger. (*Zeit. anal. Chem.*, 1900, xxxix., 627-633.)—The freshly-precipitated compounds of tannin with alkaloids can be separated from proteids by means of acetone, whilst the compounds of tannin with albumin or peptone do not yield any trace of the proteid constituent to this solvent, although some tannin is dissolved.

In forensic analysis the substance to be examined is extracted with *acidified* alcohol, and the extract evaporated. After mixing the residue with a little water and acetone, tannin is added, and the paste gently heated with acetone slightly acidified with hydrochloric acid, and filtered. The filtrate, after the addition of 10 to 20 c.c. of glycerol with a little water and 1 or 2 c.c. of hydrochloric acid, is evaporated on the water-bath, and the residue diluted with water and filtered. It can then be extracted with chloroform.

When alkaloids which are volatile with steam are present, the method formerly described by the author (ANALYST, xx., 201) of direct extraction with glycerol containing tannin is advisable. C. A. M.

The Constituents of German Rose Oil. H. Walbaum and K. Stephan. (*Berichte*, 1900, xxxiii., 2302-2308.)—According to Bertram and Gildemeister (*Journ. prak. Chem.*, 1894, xlix., 185), both Bulgarian and German rose oil consist essentially of geraniol and odourless hydrocarbons. Citronellol has also been discovered by Tiemann in the Bulgarian oil. As these two substances did not possess the odour of rose oil when mixed, the authors have examined a large quantity of German oil to discover what other substances were present. By fractional distillation, partly with steam and partly under reduced pressure, the constituents of lower boiling-point than geraniol were separated. These were refractionated under reduced pressure, yielding finally about 300 grammes of distillate boiling from 55° to 100° C. under 13 millimetres pressure, and a fraction of higher boiling-point which still contained geraniol.

The lower fraction yielded on treatment with bisulphite an aldehyde identified as nonyl aldehyde, and the residue left after the removal of this was found to contain linalool.

The fraction of the oil boiling above 100° C. at 14 millimetres pressure was treated with bisulphite, and citral was identified in the compound produced. After removal of the citral, the fraction was found to contain a small quantity of phenyl-ethyl alcohol.

Citronellol was also identified in the oil, and in the authors' opinion other odoriferous substances were probably present. C. A. M.

The Occurrence of Phenyl-ethyl Alcohol in Rose Petals and Rose Oil. H. Walbaum. (*Berichte*, 1900, xxxiii., 2299-2302.)—In a previous communication (*Berichte*, xxxiii., 1903) the author has shown that phenyl-ethyl alcohol is obtained by extracting dried rose petals, and that it is also present to a slight extent in normal rose oil. On extracting 90 kilos of the petals with ether, and distilling the residue of the extract in a current of steam, a turbid aqueous distillate was obtained, from which about 80 grammes of a brown oil could be extracted with ether. This was found to consist for the most part of phenyl-ethyl alcohol.

Since the ordinary rose oil obtained by distillation of the petals with steam never possesses the exact odour of the rose, numerous attempts have been made from time to time to extract the odoriferous constituents with petroleum spirit, carbon bisulphide, or other solvents. As in the case of the dried flowers, it was found that the freshly-plucked blossoms when extracted with petroleum ether also yielded an oil consisting principally of phenyl-ethyl alcohol. Geraniol, which is a principal constituent in the oil obtained by distillation of the fresh blossoms with steam, is only present to a small extent in the extracted oil. The author considers that this great difference between the distilled and extracted oils renders a re-examination of normal rose oil necessary.

C. A. M.

The Determination of the Acid and Saponification Values of Wax. Otto Eichorn. (*Zeit. anal. Chem.*, 1900, xxxix., 640-645.)—Amyl alcohol is recommended instead of ordinary alcohol for these determinations.

Acid Value.—About 6 grammes of the sample are heated to the boiling-point with 60 c.c. of pure amyl alcohol, and then titrated in the ordinary way. The advantage over the ordinary method is that the titration can be completed without the necessity of again heating the liquid, and any partial saponification is thus obviated. The author therefore obtained results considerably lower in this way than by the older method. Thus:

	Beeswax.		Japan Wax.	Carnauba Wax.
Amyl alcohol	22·34	19·18	19·06	9·71
Ethyl alcohol	22·8	19·60	20·4	—

Saponification Value.—About 5 grammes of the wax are heated in the water-bath with 60 c.c. of amyl alcohol and 25 c.c. of N potassium hydroxide, a blank determination being simultaneously made. The excess of alkali is titrated after thirty minutes.

The author states that in every instance the ordinary method yielded too low results in the case of the waxes and mixtures which he examined.

The following results show that the saponification by the amyl alcohol method is complete in fifteen minutes.

	Fifteen Minutes' Saponification.	Thirty Minutes' Saponification.	Fifty Minutes' Saponification.	Ninety Minutes' Saponification.
Wax No. 1	94·6 94·3 } 94·4	94·5 94·5 } 94·5	94·3 94·4 } 94·3	94·4
Mixture of wax No. 1, 75 %, and ceresin, 25 %. Value calculated on the wax ...	94·4	94·2	94·4	—
Wax No. 5	95·7 95·1 } 95·4	95·4 95·4 } 95·4	95·3 95·0 } 95·1	95·3

C. A. M.

The Analysis of Gutta-percha. H. Bornträger. (*Zeit. anal. Chem.*, 1900, xxix., 502-504.)—Crude commercial gutta-percha has the following composition, according to the investigations of different chemists: Water, 1 to 1·5; dirt (wood and soil), 3 to 5; pure gutta, 30·5 to 83·5; albane, 7·0 to 44·5; and fluavile, 3·0 to 21·0 per cent.

It is generally accepted that gutta is a hydrocarbon ($C_{10}H_{16}$), and that albane ($C_{10}H_{16}O$) and fluavile ($C_{40}H_{64}O_3$) are only oxidized gutta. The author finds that this is only true of albane, which is insoluble in cold alcohol, and which consists of three separate resinous substances. Of these a_1, which constitutes about 30 per cent. of the albane, is a light yellow resin oil, which distils at 200° C.; a_2 (about

30 per cent.) is a dark yellow oil distilling at 250° C.; and a_3 (about 40 per cent.) is a solid resin resembling colophony.

Fluavile, on the other hand, is a definite substance, which distils as a colourless resin oil at 105° C. It is soluble in cold alcohol.

Estimation of Water.—Two grammes of the finely-divided sample are dried in a weighed tube in a current of air at 100° C. until the weight becomes constant.

Estimation of Dirt.—One gramme of the substance is heated on a water-bath under a reflux condenser for twelve hours with 50 c.c. of benzene. The liquid is then filtered, and the flask and filter washed with hot benzene until the washings leave no residue on evaporation. The filter and flask are then dried at 110° C., and weighed.

Estimation of Pure Gutta.—The benzene with the resinous compound in solution is evaporated to about 50 c.c., mixed with 100 c.c. of absolute alcohol, and allowed to stand for two hours on the water-bath. The pure gutta is precipitated and separated, washed three times with hot alcohol, dried at 100° C., and weighed.

Determination of Albane and Fluavile.—These are generally estimated together by difference. For a separate determination the author evaporates the alcohol and benzene filtrate from the estimation of the gutta to 50 c.c., and leaves it to cool in a weighed basin. After twenty-four hours the supernatant liquid with the fluavile in solution is decanted, and the deposit of albane washed three times with cold alcohol, dried at 80° C., and weighed. The fluavile is found by difference, or by evaporating its alcoholic solution after removing the albane. If a determination of its three constituents be required, the dried albane may be fractionally distilled.

An analysis of a sample of crude gutta-percha by the above method gave the following results: Water, 1·5; dirt, 2·5; pure gutta, 77·5; fluavile, 6; albane a_1, 3·8; albane a_2, 3·7; and albane a_3, 5 per cent. (*cf.* ANALYST, xxi., 74, and xxii., 328).

C. A. M.

The Determination of Proteids in Feeding Stuffs. H. Schjerning. (*Zeit. anal. Chem.*, 1900, xxxix., 633-639.)—As the author has found that uranium acetate precipitates true peptones, whilst Stutzer's reagent does not, he has made experiments to determine whether peptones are actually present in feeding stuffs.

He has obtained the following results in his analysis of various samples by the method previously described (ANALYST, xxiii., 326):

	Molasses Fodder.	Cotton-seed Cake.	Blood-molasses Fodder.	Sunflower Cake.	Rape-seed Cake.	Palm-oil Cake.
Albumins	9·8	2·0	0	51·8	49·1	0
Denuclein	10·7	58·0	14·3	10·3	5·0	31·2
Propeptone	1·8	8·0	4·8	5·1	11·7	6·2
Peptone	1·8	0	0	0	3·3	0

From these figures, the conclusion is arrived at that peptones are not present in ordinary feeding stuffs, or, if so, only in very minute quantities; and that, therefore, Stutzer's reagent and uranium acetate should precipitate practically the same proportion of nitrogen. This was confirmed experimentally.

In the method finally recommended, 0·5 to 1 gramme of the sample is mixed with 100 c.c. of water, and left for twenty hours, with frequent agitation. The contents of the beaker are then heated to 50° C. in the water-bath, and 20 to 40 c.c. of a saturated solution of uranium acetate introduced. After standing for thirty minutes at the same temperature, the precipitate is filtered off, washed with a cold 1 to 2 per cent. solution of uranium acetate, boiled with 50 c.c. of magnesia milk (11 grammes MgO in 2 litres of water) to expel ammonia, and evaporated *nearly* to dryness. The nitrogen in the residue is then determined in the usual way, a correction of 0·1 c.c. $\frac{N}{10}$ acid being made for each 100 c.c. of filtrate and washings.

In the following table of results thus obtained with the different feeding stuffs, A represents the nitrogen precipitated by Stutzer's reagent; B by uranium acetate, without subsequent boiling with magnesia; and C by uranium acetate and evaporation with magnesia:

Nitrogen per Cent. as	Barley.		Maize.		Molasses Fodder.		Cotton-seed Cake.			Blood-molasses Fodder.			Sunflower Cake.			Rape-seed Cake.			Palm-oil Cake.		
	A	B	A	B	A	B	A	B	C	A	B	C	A	B	C	A	B	C	A	B	C
Proteids ...	1·425	1·423	1·198	1·211	1·119	1·121	6·505	6·379	6·385	1·941	1·991	1·934	5·794	5·640	5·650	4·882	4·943	4·853	2·780	2·790	2·736
Other N substances	0·075	0·077	0·081	0·068	0·817	0·815	0·260	0·386	0·380	0·506	0·456	0·513	0·267	0·421	0·411	0·345	0·284	0·374	0·170	0·160	0·214
Total ...	1·500		1·279		1·936		6·765			2·447			6·061			5·227			2·950		
Allowable error ..	0·012		0·012		0·010		0·026			0·028			0·019			0·030			0·016		

C. A M.

INORGANIC ANALYSIS.

Volumetric Method for the Estimation of Copper. **S. W. Parr.** (*Journ. Amer. Chem. Soc.*, 1900, 652.)—This is a modification of the methods proposed by Wallace and Smith (ANALYST, 1898, 50) and Meade (*ibid.*, 332). A solution containing about 0·04 gramme of copper and 1 to 2 per cent. of nitric acid is reduced by adding a few c.c. of strong sulphurous acid or of 10 per cent. sodium sulphite, and heating. Two c.c. of a 10 per cent. solution of an alkaline thiocyanate are then added. The precipitated cuprous thiocyanate is filtered off on an asbestos filter, and thoroughly washed, until the washings do not decolorise permanganate. The wad is placed in a beaker, 2 or 3 c.c. of a 10 per cent. caustic soda solution are added, and the whole is warmed to 70°. Decinormal permanganate is now run in until the yellow colour changes to green. Dilute sulphuric acid (1 : 5) is added until the solution becomes clear, the solution is warmed to 60° or 70°, and permanganate is run in until the solution becomes pink.

The oxidation may be represented by the equation

$$10CuCNS + 7K_2Mn_2O_8 + 21H_2SO_4 = 7K_2SO_4 + 14MnSO_4 + 10HCN + 10CuSO_4 + 16H_2O.$$

From which it will be seen that 1 c.c. $\frac{N}{10}$ permanganate is equivalent to $\frac{63{\cdot}3}{7}$ milligrammes of copper.

A. M.

Determination of Carbon in Ferrochrome. A. A. Blair. (*Journ. Amer. Chem. Soc.*, xxii., 719.)—Twenty-five grammes of potassium bisulphate are fused in a platinum boat 150 millimetres long and 25 millimetres wide. When cold, 1 gramme of the finely-ground sample is spread over the surface, the boat placed in another larger one, into which it fits, and a platinum cover put over it, which prevents any splashing of the bisulphate into the tube. The boat is placed in a platinum tube 400 millimetres long and 30 millimetres in diameter, and closed by a ground joint at the rear. Condensation of sulphuric acid around the joint is prevented by placing two plugs of pumice wrapped in platinum foil between it and the boat. The forward end of the tube is contracted and filled with platinized asbestos to oxidize sulphurous acid, and then still further contracted, bent downwards, and fused to a glass U-tube filled with glass beads.

The combustion is made in oxygen, purified as usual, the heating being started at the contracted end of the tube. The products of combustion, after leaving the U-tube, pass through two flasks heated by a burner and filled with a solution of 150 grammes chromic acid and 300 c.c. sulphuric acid per litre. They then pass through a U-tube filled with glass beads, serving as condenser, next through a tube containing pumice saturated with chromic acid, and then through a calcium chloride tube, after which they enter the absorption apparatus. A gasometer connected to the latter by a guard tube serves to relieve the pressure, which would otherwise be considerable.

As the current of gas and the heating must be very slow, a combustion requires about two hours and a half. After washing the boats and cover they are ready for another determination. Duplicate analyses are stated to agree within 0·01 per cent.

A. G. L.

The Separation of Tungsten Trioxide from Molybdenum Trioxide. Max J. Ruegenberg and Edgar F. Smith. (*Journ. Amer. Chem. Soc.*, xxii., 772.)—The method is based on the fact that tungstic acid is insoluble in concentrated or dilute sulphuric acid, whilst molybdenum trioxide is readily soluble (*Journ. Amer. Chem. Soc.*, xix., 242). Sulphuric acid of specific gravity 1·378 was found to give the best results. The results of six experiments are given, which show the method to be very exact. Another set of experiments, in which the tungsten trioxide (0·2 to 1·3 gramme) was mixed with 5 grammes ferric hydroxide before treating with sulphuric acid showed that the presence of iron is without influence on the solubility of tungsten trioxide.

A. G. L.

Volumetric Determination of Thallium. H. Marshall. (*Journ. Soc. Chem. Ind.*, 1900, 994.)—A weighed quantity of the substance containing thallium in the form of thallous salt is heated with potassium bromide, hydrochloric acid, and a measured excess of a standard solution of sodium or potassium bromate. The bromine formed is boiled off, and condensed in a condenser, the delivery tube of which dips into potassium iodide solution. Part of the bromate is used up in oxidizing the thallous to thallic salt—

$$3TlCl + NaBrO_3 + 6HCl = 3TlCl_3 + NaBr + 3H_2O;$$

the remainder is determined by titrating the iodine set free with thiosulphate solution. The author has devised an apparatus entirely of glass for carrying out the distillation. A. M.

Iodometric Estimation of Chromic Acid. K. Seubert and A. Henke. (*Zeit. f. angew. Chem.*, 1900, 1147.)—The method depends upon the reaction:

$$K_2Cr_2O_7 + 6KI + 7H_2SO_4 = 4K_2SO_4 + Cr_2(SO_4)_3 + 7H_2O + 6I.$$

Under ordinary circumstances the action takes considerable time. The authors have made an exhaustive investigation of its rate of progress when the different bodies are present in different quantities. Increasing the proportion of acid accelerates the reaction more than increasing the proportion of potassium iodide does; dilution greatly retards it. The following are convenient proportions to use: Bichromate, 0·05 gramme; potassium iodide, 0·5 gramme; sulphuric acid, 1·8 gramme; total volume, 100 c.c. If less than 0·05 gramme of bichromate be present, the other quantities should still be kept the same. If there be more bichromate, the iodide and acid should be proportionately increased without adding more water, unless there be more than 0·25 gramme. The reaction is complete in about six minutes. The liquid should then be diluted and titrated with thiosulphate solution, using starch as indicator. A. M.

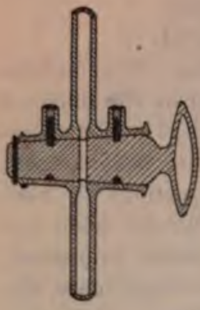
Determination of Phosphoric Acid as Phosphomolybdic Anhydride. H. C. Sherman and H. St. J. Hyde. (*Journ. Amer. Chem. Soc.*, 1900, 652.)—The authors have examined Woy's method (ANALYST, 1897, 250 and 333), in which the molybdate precipitate is ignited, and weighed as $24MoO_3,P_2O_5$, and find the method to give results which agree exactly with those obtained by the molybdate-magnesia method. The second precipitation recommended by Woy is, however, quite unnecessary, equally good results being obtained with only one precipitation. A. M.

The Preparation of Standard Acid. C. L. Higgins. (*Journ. Soc. Chem. Ind.*, 1900, xix., 958.)—The author finds that sodium carbonate as a base for alkalimetry is unsatisfactory, for it is not possible to convert bicarbonate entirely into carbonate without forming some caustic soda. He finds, however, that G. T. Moody's method (*Journ. Chem. Soc.*, 1898, 68) gives very accurate results. Pure dry hydrogen chloride gas is passed into a weighed absorption bulb containing water. The bulb is made entirely of glass, and is fitted with a stopcock and a calcium chloride tube. The increase of weight of this bulb gives exactly the weight of hydrogen chloride absorbed. A. M.

Electrolysis of Copper Sulphate as a Basis for Acidimetry. C. A. Cohn. (*Journ. Soc. Chem. Ind.*, 1900, 962.)—If a neutral solution of pure copper sulphate be electrolysed in the manner usual in copper determinations, copper is deposited, and the equivalent quantity of sulphuric acid is set free. From the weight of copper deposited the exact quantity of acid can be calculated, and the latter can be used as a basis for acidimetry. The results obtained agree exactly with those given by the hydrogen chloride method (see preceding extract).

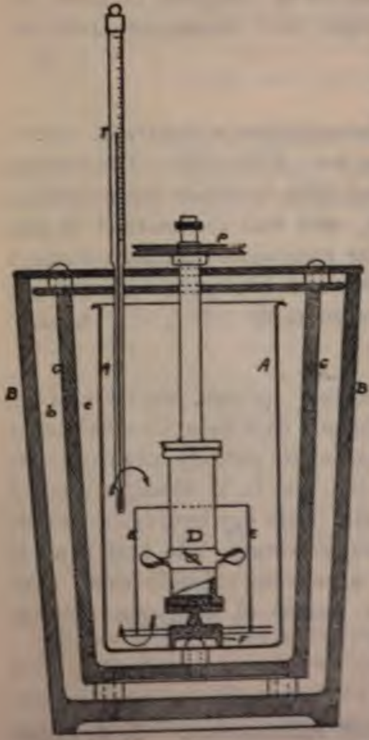
A. M.

APPARATUS.



Gastight Stopcock. H. Göckel. (*Zeits. f. angew. Chem.*, 1900, 961.)—The stopcock is designed so that it may be used in any position. The key has two grooves cut in it, one on each side of the bore. The mantle has two small tubes running through it corresponding with the grooves. The grooves and tubes are filled with mercury, and then the tubes are corked and sealed. To prevent the key coming out, the end is made to project somewhat, and a pin is run through it as shown.

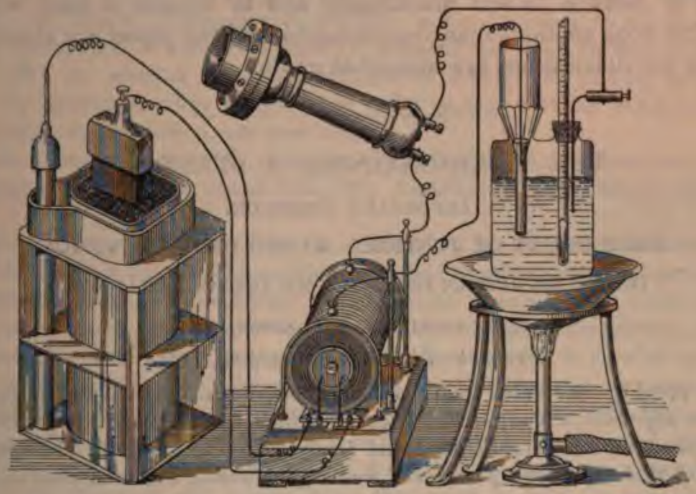
A. M.



New Coal Calorimeter. S. W. Parr. (*Jour. Amer. Chem. Soc.*, 1900, 646.)—The coal is ground up to pass a 100-mesh sieve, dried at 105° C., and 1 gramme is weighed out and put into the cartridge D; 16 to 18 grammes of sodium peroxide are added, the top is screwed on, and the whole is shaken to thoroughly mix the contents. It is then put into the insulated can A, 2 litres of water are added, the lid of the can is replaced, and the pulley P is connected with a motor and rotated at 50 to 100 revolutions per minute. When the temperature is constant, a hot wire is dropped down the stem of the cartridge, which is provided with a valve to close again after the wire has passed. The maximum temperature is noted, and the heat of combustion C is calculated from the formula $C = (t - {\cdot}012^\circ) \times w \times 0{\cdot}73$, where t is the rise of temperature observed, w is the water employed, ·012° C. is the rise caused by the hot wire ($\frac{1}{2}$ inch of No. 12 copper wire), and 0·73 is the necessary factor which has been determined by a special series of experiments.

A. M.

Determination of Correct Melting-points, II. Thomas Tyrer and Albert Levy. (Notes from the Laboratory of the Stirling Chemical Works, July, 1900.)—*Levy's Acoustical Method.*—The apparatus used consists of a small battery, a Ruhmkorff's coil, a Chrystomanos' beaker (*Ber.*, 33, 1093), and a telephone. An alternating current is used to do away with the secondary current which may be formed by the electrolysis of the substance. The internal diameter of the end of the tube lodged in the mercury is 4 millimetres. The powdered substance is pressed into this to a height of 1·5 millimetres, by means of a metal rod of diameter 3·5 millimetres, and then mercury is poured in to a height of 20 to 30 millimetres, if necessary, after the tube has been fixed in the beaker. As soon as the substance melts the mercury falls, contact is made, and the noise of the commutator produces a loud sound in the telephone. The method gives good results. A. S. L.



REVIEW.

Bacteria, especially as they are related to the Economy of Nature, to Industrial Processes, and to the Public Health. By George Newman, M.D. Second edition, 1900. Pp. 381. Twenty-three plates and sixty other illustrations. London : John Murray (The Progressive Science Series). Large 8vo. Price 6s.

This book is a popular scientific statement of our present knowledge of bacteria. It is brought well up to date, and gives the results of many of the latest researches. It does not profess to be a practical handbook, although its pages contain many valuable technical hints relating to the preparation and uses of nutrient media, methods of microscopical staining, etc. A chapter is devoted to the bacteriological examination of water, and to the identification of many specific organisms found in water. We are glad to note the emphasis laid upon the necessity of *both* chemical and bacteriological examination of water intended for drinking purposes. The danger of domestic filters other than those of the Pasteur-Chamberland pattern is pointed out. The bacteriological treatment of sewage is dealt with pretty thoroughly, the work of Jordan, Dibdin, and Cameron in this direction being referred to; and the methods of treatment adopted in London, Sutton, Exeter, and Manchester are described. The bacteriology of milk and milk products, and the dissemination of diseases (especially tuberculosis) through these substances, also receive considerable attention, the advantages of pasteurization being pointed out. The subject of bacteriology in relation to soil nitrification and to disease is dealt with in other chapters. The book contains many useful tables, and the plates are excellent, though one or two of the illustrations are somewhat crude. J. F. H. G.

THE PHARMACOPŒIA A STANDARD.

IMPORTANT DECISION.

HIGH COURT OF JUSTICE. KING'S BENCH DIVISION.

(Before MR. JUSTICE BRUCE and MR. JUSTICE PHILLIMORE.)

DICKINS *v.* RANDERSON.

(*From the Times of January* 30, 1901.)

THIS was an appeal by case stated from the decision of Justices of the West Riding of Yorkshire convicting the appellant, a pharmaceutical chemist, upon an information preferred against him by the respondent, an Inspector of Food and Drugs, charging him with unlawfully selling, to the prejudice of the respondent, 2 ounces of mercury ointment, which on analysis was found to be deficient in mercury.

At the hearing of the information, the following facts were proved or admitted: On May 14, 1900, the respondent went to the appellant's premises and asked for 2 ounces of mercury ointment. Ointment was supplied to him in a small wooden box, the lid of which was labelled "The Ointment—Mercurial Poison," for which he paid fourpence. Upon analysis, the substance supplied was found to contain the parts as follows: mercury, 12·5 per cent.; lard and suet, 87·5 per cent. The analyst added to his analysis a statement that mercury ointment, according to the British Pharmacopœia, contained 48·5 per cent. of mercury, and the mercurial activity of the sample would only be one-fourth that of an ointment prepared according to the British Pharmacopœia. It was contended by the respondent that, as the ointment supplied was not according to the standard of the British Pharmacopœia, it was not of the "nature, substance, and quality demanded" within the meaning of section 6 of the Sale of Food and Drugs Act, 1875. It was contended on behalf of the appellant that the proceedings would not lie under section 6 of the Act, but ought to have been commenced under section 7, the article in question being a "compounded drug." This objection having been overruled, the appellant gave evidence that the ointment supplied was a dilute preparation of mercury ointment well known to and supplied by all retail chemists, and such as would be usually supplied to a person asking for "mercury ointment." He stated that out of hundreds of sales of that commodity which he had made he had never supplied it according to the standard of the British Pharmacopœia for human application, except when making up the prescription of a medical practitioner, for the reason that he considered that the frequent use thereof by a person of delicate constitution might probably set up mercurial poisoning, from which death might ensue. Both the dilute and the standard preparation were kept in stock by the appellant's employers at the premises in question. The dilute preparation was known to the public by a variety of names, and it was usually asked for under the name of "blue ointment." Two other chemists gave similar evidence. It was contended on behalf of the appellant that he was not in law required to sell mercury ointment to a purchaser in accordance with the standard quality prescribed by the British Pharmacopœia, except when specifically asked by the purchaser to do so, or when making up the prescription of a medical practitioner; and that accordingly it was within the discretion of the appellant to supply the diluted preparation in all cases in which he thought fit to do so, and that he had therefore committed no offence under section 6. The Justices found that the commodity sold was not of the nature, substance, and quality of the article demanded by the purchaser, and held that the appellant was bound to supply the commodity according to the formulary prescribed in the British Pharmacopœia, whether expressly asked to do so or not. The questions for the opinion of the Court were (1) whether the appellant was so bound as last aforesaid or not; (2) whether there was evidence to support the finding of the Justices; (3) whether the information was properly laid under section 6 of the Food and Drugs Act, 1875; (4) whether the conviction was good in law.

Mr. C. A. **Russell**, K.C. (with whom was Mr. Compston), appeared for the appellant; Mr. **Danckwerts**, K.C. (with whom were Mr. Roskill and Mr. Caine), represented the respondent.

The following judgment of the Court was delivered by Mr. Justice **Phillimore**: We think that this conviction must stand. The appellant professed to sell mercurial or mercury ointment. Mercury ointment is a drug in the British Pharmacopœia, known by that name, and having a certain fixed ratio of mercury to unguent. The appellant was asked for mercury ointment, and should have sold the drug in the Pharmacopœia, or, if he was going to do what he in fact did—namely, sell an ointment in which the mercury was about one-quarter strength—he should, if he may lawfully sell such a drug (see section 15 of the Pharmacy Act, 1868), have explained that he was selling a weaker or diluted drug, and have so named it. He acted under no sordid motive, but, nevertheless, he was in the habit of selling to his customers something which was not of the nature, substance, and quality which they must be taken to have demanded, and which certainly the purchaser in this case demanded. It is said that the appellant sold the mercury ointment of commerce, and that for commercial purposes there is a standard different from that of the Pharmacopœia. Having regard to section 15 of the Pharmacy Act, 1868 (31 and 32 Vict., c. 121), that is unlikely, though perhaps possible. But he failed altogether to prove that there was any commercial standard for this article different from that of the Pharmacopœia. He did not even attempt to prove it. What he attempted to prove was that there were two commercial standards of wide difference, a thing in itself unreasonable, while he stated that he kept both standards in his shop, and sold according to one standard or the other, according as the customer brought or did not bring a prescription from a medical man. He might almost as well have kept two weights and two measures. The practical mischief of such a practice was pointed out in the argument. If there be any danger on the other side, it would be avoided by the simple expedient of having (if he lawfully may) two drugs, calling them, as in fact they are, the strong one "mercury ointment," the weak "dilute mercury ointment," and recommending the latter to any customer whom he might think unskilled and likely to use the proper drug in a dangerous manner. The cases of White *v.* Bywater (L.R. 19, Q.B.D., 582) and Beardsley *v.* Walter (L.R., 1900, 2 Q.B.) almost, if not quite, conclude the matter. But without them we should have arrived at the same result upon the construction of section 6 of the Sale of Food and Drugs Act, 1875 (38 and 39 Vict., c. 63). They establish the fact, and we should have thought without their assistance, that if a drug to be found in the Pharmacopœia is asked for, this drug must be supplied, and that if it is not sold with the ingredients and in the proportions prescribed by the Pharmacopœia there is at least *primâ facie* evidence that what is sold is not of the nature, substance, and quality which was demanded. It was argued that, as the drug in question was a compounded drug, proceedings ought not to have been taken under the sixth section of the Sale of Food and Drugs Act, but under the seventh, and for this purpose some observations of Mr. Justice Wright's in Houghton *v.* Taplin (13 the *Times* Law Reports, 386) were invoked. We think that those observations have been misunderstood. In any case, it is quite clear from the definition clause that a compounded drug is none the less a drug, which shows that proceedings can be laid under section 6, though it may be that in the case of a compounded drug they can also be laid under section 7. We wish to add a few words with reference to the exception contained in sub-section 3 of section 6. We entertain no doubt that the appellant sold, to the prejudice of the purchaser, a drug which was not of the quality of the article demanded by such purchaser, and so contravened the provisions of the earlier part of section 6. But the difficulty, we have felt, arises from the provision that an offence shall not be deemed to be committed under that section where the drug is compounded as in the Act mentioned. It was argued by counsel for the appellant that because the article in question was a compounded drug the sale of it fell within the exception in sub-section 3 of section 6, and constituted an offence, not under section 6, but only under section 7. We think it probably did constitute an offence under section 7, but we think it did also constitute an offence under section 6. The offence in subsection 3 of section 6 does not apply to all compounded drugs, but only to a drug compounded as in the Act mentioned. It is very difficult to understand the meaning of the words "as in this Act mentioned." We find no provisions in the Act to which the words can apply. But it seems to us it is impossible to reject the words. We cannot read the exception in sub-section 3 as if it were not qualified by the concluding words. But, although it is difficult to say what is the exact meaning of the words, this, we think, is clear—that no possible meaning that can be given to the words can apply to the ointment in the present case. It was certainly not compounded as in the Act mentioned. The sale of it, therefore, does not fall within the exception.

Conviction affirmed.

INSTITUTE OF CHEMISTRY OF GREAT BRITAIN AND IRELAND.

THE following is a list of the names of the candidates who passed the examination of the Institute of Chemistry held in January, 1901:

INTERMEDIATE EXAMINATION.—Evans, J., King's College, London, and Pharmaceutical Society. Finlow, R. S., University College of North Wales, Bangor. Hinks, E., King's College, London. Handcock, W. A., Finsbury Technical College, London. Northall-Laurie, D., King's College, London, and under E. Jackson, Esq., F.I.C. Steedman, G., Glasgow and West of Scotland Technical College.

FINAL A.I.C. EXAMINATION.—Branch "A" (*Mineral Chemistry*): Austin, J. H., Birmingham University. Clark, R. M., B.Sc. (Glasg.), Glasgow University. Cotterill, J. W., Birmingham University (for Fellowship). Cunliffe, F., Owens College, Manchester. Dick, W. D., King's College, London. Gray, A., Glasgow and West of Scotland Technical College. Lowson, W., Yorkshire College, Leeds. Robertson, A. J., University College, Dundee, and under R. R. Tatlock, Esq., F.I.C. Branch "B" (*Metallurgical Chemistry*): Bowen, W., Birmingham University. Branch "D" (*Organic Chemistry*): Carter, R. J., B.A. (Oxon.), Oxford University. Etheridge, A. T., B.Sc. (Lond.), Birmingham University. Lloyd, J. A., Birmingham University. Rogers, J., Glasgow and West of Scotland Technical College. Taylor, W. H., Finsbury Technical College, London. Branch "E" (*Analysis of Food and Drugs, not including Examination in Therapeutics, Pharmacology and Microscopy*): Bayly, H. G., King's College, London. Denny, E. J., A.C.G.I., Central Technical College, London, and under F. Sutton, Esq., F.I.C. Hewitt, H. D., Finsbury Technical College, London, and under A. C. Chapman, Esq., F.I.C. Johnstone, J. H., B.Sc. (Vict.), Owens College, Manchester. Branch "E" (*Analysis of Food and Drugs, including an Examination in Therapeutics, Pharmacology and Microscopy*): Chapman, E. M., Pharmaceutical Society and King's College, London. Dewhirst., J. A., Yorkshire College, Leeds, and Pharmaceutical Society. Mitchell, A. H., B.Sc. (Lond.), Owens College, Manchester, and University College, London (for Fellowship). Pinchin, E. A., B.Sc. (Lond.), Royal College of Science, London.

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THE ANALYST.

MARCH, 1901.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE annual general meeting of the Society was held on Tuesday afternoon, February 5, in the Chemical Society's Rooms, Burlington House. The President (Mr. W. W. Fisher, M.A.) occupied the chair.

Before the business of the meeting was proceeded with, the PRESIDENT said that, on account of the recent death of the Queen, the annual dinner of the Society, which was to have taken place on that evening, had been postponed. The Council on the Society's behalf had sent a message of condolence expressing sincere sorrow at the heavy loss sustained by the nation, and tendering their loyal sympathy with His Majesty the King and the Royal Family in their deep affliction.

The minutes of the previous meeting were then read and confirmed.

The PRESIDENT announced that Dr. T. E. Thorpe, C.B., F.R.S., Principal Chemist of the Government Laboratory, had been nominated by the Council for election as an honorary member of the Society.

Messrs. Percy Henry Carpenter and Samuel Russell Trotman, M.A., were elected members of the Society.

The HON. TREASURER (Mr. E. W. Voelcker, A.R.S.M.) presented his annual report.

On the motion of Mr. SMETHAM, seconded by Mr. EKINS, the report was adopted, and a vote of thanks accorded to the Hon. Treasurer.

The HON. TREASURER proposed, and Mr. ARCHBUTT seconded, a vote of thanks to the Auditors, which was carried unanimously.

On the proposition of the PRESIDENT, a vote of thanks was passed to the Chemical Society for the permission to use their rooms during the past year.

The PRESIDENT then delivered his retiring address.

On the proposition of Mr. ALLEN, a vote of thanks was accorded to the President for his address; and it was resolved that, with the President's permission, the address should be published in THE ANALYST.

The Officers and Council for 1901 were declared to be elected in accordance with the nominations made by the existing Council.

Dr. Voelcker proposed, and Dr. Dyer seconded, a vote of thanks to Mr. Fisher for the services which he had rendered to the Society as its President. The proposition was carried unanimously.

Mr. Fisher, having responded, introduced the newly-elected President, Dr. J. Augustus Voelcker, in whose favour he then vacated the chair.

Dr. Voelcker, on taking the chair, said that he had received a letter from Dr. Vieth expressing his great regret that he was unable to be present at the annual meeting.

The proceedings then terminated.

OFFICERS AND MEMBERS OF COUNCIL FOR 1901.

President.—J. Augustus Voelcker, M.A., B.Sc., Ph.D.

Past-Presidents.—M. A. Adams, F.R.C.S., Alfred H. Allen, Sir Charles A. Cameron, C.B., M.D., F.R.C.S., A. Dupré, Ph.D., F.R.S., Bernard Dyer, D.Sc., W. W. Fisher, M.A., Otto Hehner, Alfred Hill, M.D., J. Muter, M.A., Ph.D., Thos. Stevenson, M.D., F.R.C.P.

Vice-Presidents.—Wm. Chattaway, H. Leffmann, M.D., Francis Sutton.

Hon. Treasurer.—E. W. Voelcker, A.R.S.M.

Hon. Secretaries.—E. J. Bevan, Alfred C. Chapman.

Other Members of Council.—L. Archbutt, A. Ashby, M.B., F.R.C.S., Julian L. Baker, Bertram Blount, W. J. Dibdin, Arthur E. Ekins, G. Embrey, Thomas Fairley, J. H. B. Jenkins, Percy A. E. Richards, H. Droop Richmond, Clarence A. Seyler, B.Sc.

The monthly meeting of the Society was held on Tuesday evening, February 5, in the Chemical Society's Rooms, Burlington House. The President (Dr. J. Augustus Voelcker, M.A., B.Sc.) occupied the chair.

The minutes of the annual meeting, held at an earlier hour on the same date, were read and confirmed.

Certificates of proposal for election in favour of Messrs. A. L. H. Garside, R. G. Grimwood, J. C. Umney, and H. Rowley, were read for the second time, and certificates in favour of Mr. Henry Wippell Gadd, analyst to Messrs. Evans, Gadd and Co., Exeter, and of Mr. Albert Henry Mitchell, B.Sc. (Lond.), F.I.C., Public Analyst for the Borough of Tiverton, were read for the first time.

The following papers were read: "On the Authority of the British Pharmacopœia as a Standard under the Sale of Food and Drugs Act," by Alfred H. Allen; "The Physical State in which Fat exists in Cream," by H. Droop Richmond and Sylvester O. Richmond; "On the Composition of Dutch Butter," by John Clark, Ph.D.; and a "Note on the Proximate Analysis of Cloves," by A. McGill, B.A.

Dr. Schidrowitz exhibited a special gas-burner for use in the estimation of very small quantities of copper, and also some small pieces of apparatus for use in the application of Gutzeit's test for arsenic.

ADDRESS OF THE RETIRING PRESIDENT.

(*Delivered at the Meeting, February* 5, 1901.)

It is fitting, before we commence the business of to-day, that I should explain the course of action taken by the Council at the time of national mourning for the death of the Queen. In common with our fellow-countrymen and fellow-subjects, we have shared the sorrow caused by this event, and it would have been more consonant with our personal feelings and wishes if this annual meeting had been put off.

At a meeting of your Council, however, it was decided that, although the dinner must rightly and properly be given up, yet, in view of the necessary elections of officers and Council for the coming year, grave difficulties might arise from any postponement, and it would therefore be best to meet for the transaction of the necessary business of the Society for the coming year.

The Council, on your behalf, have sent a message of condolence expressing their sincere sorrow at the heavy loss sustained by the nation, and tendering their loyal sympathy to His Majesty the King and the Royal Family in their deep affliction.

At the end of my term of office, it is a great pleasure to look back upon a period of prosperity, and to have witnessed a period of active growth and development in our Society. We had a large accession of members under the new rules, amounting to about forty in 1899 (including associates becoming members), and in the year just closed another twenty have joined our body. No member has resigned, and our total stood at 270 at the end of the year. We have to regret the loss of two by death—Mr. Jarmain, an old member, one of our seniors, a notice of whom appeared in the February ANALYST; and a younger man, Mr. Pearmain, whose more recent appearance among us made many better acquainted with him, and all who knew him must deplore his untimely loss. In the present year, moreover, we have suffered the loss by death of another distinguished member, Dr. Sedgwick Saunders, who occupied the official position of Medical Officer of Health as well as Public Analyst for the City of London.

The Society has not only increased in numbers, but in vitality, and the interest in its proceedings is shown by the large attendance of members at our evening meetings, when topics of great importance have frequently been discussed to the advantage and enlightenment of many of us.

The increase in numbers, not accompanied by much growth of expenses, has left our finances at the close of the year in a sound, and to our Hon. Treasurer, I believe, satisfactory, condition. Our renewed thanks are due to him for kindly consenting again to control our exchequer in the coming year.

We must also congratulate our Editor on completing the twenty-fifth volume of THE ANALYST, which, I think, fully maintains in the variety and importance of the papers it contains the reputation and character of the earlier volumes. I learn from Dr. Sykes that there is a substantial increase in the amount received from subscribers, and the cost to the Society is reduced accordingly; and no doubt THE ANALYST will continually increase in value and interest. I may fairly say this is the only journal of its kind at present in England, and that it is therefore incumbent on all our members to maintain its character and influence, and help to keep it abreast of the latest advances in knowledge.

The papers read in 1900 before the Society of Public Analysts were as follows:

February 7.—"Note on the Separation of Oleic Acid from other Fatty Acids." By J. Lewkowitsch, Ph.D.

"The Analysis of a Sample of Treacle and a Sample of so-called 'Golden Syrup.'" By C. G. Matthews and A. Hyde Parker.

"A Note on Sour Milk." By H. Droop Richmond and J. B. P. Harrison.

March 7.—"The Determination of Carbon and Sulphur in Steel." By Bertram Blount.

"Maize Oil." By Rowland Williams.

"On the Assay of Creosote." By A. D. Hall, M.A.

April 4.—"The Influence of Temperature and Concentration on the Saline Constituents of Boiler Waters." By Cecil H. Cribb, B.Sc.

"An Improved Absorption Apparatus for Use in the Analysis of Essential Oils." By Alfred C. Chapman and Herbert E. Burgess.

"The Composition of Danish Butters." By Harold Faber.

May 2.—"The Composition of Milk and Milk Products." By H. Droop Richmond.

"A New Colour Reaction for Citral and Certain other Aromatic Compounds." By Herbert E. Burgess.

"The Detection of Zirconia in the Presence of Thoria." By Otto Hehner.

June 6.—"The Determination of Oxygen in Copper by Ignition in Hydrogen." By L. Archbutt.

"Uniformity in Soil Analyses." By A. D. Hall, M.A.

"The Adulteration of Wheaten Flour with Maize." By G. Embrey.

"A New Colour Reaction for Distinguishing between Certain Isomeric Allyl and Propenyl Phenols." By Alfred C. Chapman.

November 7.—"The Determination of the Available Extract of Malt." By Lawrence Briant.

"Notes on Certain British Pharmacopœia Tests." By C. G. Moor, M.A., and Martin Priest.

"Report on the Determination of Butter-fat in Margarine." By the Committee.

December 5.—"The Examination of Extract of Malt." By Walter J. Sykes, M.D., and C. A. Mitchell, B.A.

"Note on the Estimation of Glycerol." By J. Lewkowitsch, Ph.D.

"The Examination of Varnish Resins." By J. Lewkowitsch, Ph.D.

"Barium in the Spring Water of Boston Spa." By Percy A. E. Richards.

"The Analysis of Samarskite." By Arthur G. Levy.

"The Testing for Arsenic in Beer." By Alfred C. Chapman.

Besides which the following original contributions have been received:

"The Relation between the Specific Gravity, Fat, and Solids-not-Fat in Milk." By Norman Leonard, B.Sc., F.I.C.

"The Polarimetric Estimation of Camphor in Camphorated Oil." By Norman Leonard and H. Metcalfe Smith, F.I.C.

The reprinting of extracts from an Order of the Local Government Board on the Qualifications of Public Analysts brought to our notice an important public document, and I would remark that the Order affords evidence of the desire of the Board to insure a high professional standard in all appointments, which cannot but assist in keeping up the dignity and character of our office.

Referring to the work done by Public Analysts under the Food and Drugs Acts, I find that in 1899, the latest date for which statistics are published, upwards of 53,000 samples were examined, an increase of 3,500 above the previous year, and when the returns for 1900 are available, I have no doubt that a further considerable enlargement will have taken place. Within my personal experience the action of the Board of Agriculture under the powers of the new Act has in certain districts caused a larger number of samples to be purchased, and the recent circular of the Local Government Board calling attention to the possible presence of arsenic in beer, jams, preserves, and syrups has led Local Authorities to use their powers under the Food and Drugs Acts, and additional inspectors have been appointed to make purchases of these and similar products.

The proportion of articles reported against was 5,000, or little more than 9 per cent. of the whole number, and when more numerous purchases are made the proportion is likely to become still smaller.

Milk and butter, as of old, are the articles most frequently bought (32,000 samples), and were adulterated to the extent of about 10 per cent.

The adulteration of spirits, nearly always by adding water, is still too frequent, as one-eighth of the samples examined were condemned. On the other hand, it is satisfactory to notice that bread, flour, and oatmeal are now seldom adulterated, since only 3 samples of bread out of nearly 600 were reported against, and only 9 of flour and 2 of oatmeal out of about 1,000 samples.

Tea also is remarkably free from objection, owing doubtless to the Customs inspection of imports; and general groceries, such as arrowroot, rice, tapioca, sago, and pickles, show a very low percentage of samples of unsatisfactory character.

The proportion of coffee samples condemned is this year smaller than usual, owing to the more general practice of mixtures of coffee with chicory being put up in tins duly labelled. There are still, however, a large number of articles of food, etc., which are seldom or never purchased for examination, and it appears desirable in respect of many that occasional samples should be taken.

The highest proportion of adulteration occurs in the case of drugs, as nearly 18 per cent. of these were found to be adulterated or not of the required composition. Certain popular drugs or medicines are frequently sold in retail shops, and appear peculiarly liable to adulteration. Pharmaceutical chemists, by reason of their technical knowledge, can protect themselves against fraudulent practices, but the ordinary shopkeeper is sometimes punished for the sale of an adulterated drug sold in entire innocence. Some prosecutions of the wholesale firms have, I believe, had a wholesome effect in checking the sale of improperly-prepared drugs, and if systematic purchases of such articles are made continually, the high proportion of samples now complained of will be reduced.

A glance at the list of articles drawn up by your Council for the Preservatives

Committee will enforce this point, and perhaps if this list appeared in THE ANALYST, it might afford suggestions to those who make purchases under the provisions of the Acts.

An interesting matter in connection with this review of the year's work is the small number of samples in regard to which the opinion of the Public Analyst was disputed, and a reserved sample sent to the Government chemist for analysis. I have said that nearly 5,000 samples were condemned, and in only seventy-one instances was the reference to the Government chemist demanded, with the result that the Public Analyst was supported in fifty-seven cases, while the Government chemist disagreed in fourteen, half of which were milk samples.

Although we must not claim absolute infallibility, we can look upon the proportion of 99·974 per cent. of the theoretical possibility of accuracy as a good working approach to it. Possibly some of you may further dissent in respect of some of the fourteen cases in which the Government chemist "disagreed."

We have now had a year's experience of the working of the amended Food and Drugs Act, which in the main has proved, in my opinion, a success. The regulations as to the division of samples work smoothly, and really are less troublesome to the inspectors; but there is a difficulty in some cases where articles are very small, and on division give an inadequate quantity for analysis. Some attempt to secure greater uniformity as regards packages and labels is desirable. Fears were expressed that reference samples would increase when either party in a prosecution had the right to ask for an analysis of the reserved sample, but so far this does not seem to have happened, although the exact figures are not yet available. The provision also that the certificate of a Public Analyst upon the vendor's sample shall be admitted as evidence (an eminently just provision) has not given rise, as far as I am aware, to any difficulties, and in some cases appears to have saved disputes and a possible appeal to the Government laboratory. As I have pointed out previously, such certificates are given by responsible men after conscientious consideration, and are likely to assist rather than hinder the course of justice. It usually happens, moreover, that the analysis of the vendor's sample confirms the first report of the Public Analyst.

The question of the authority of the British Pharmacopœia has lately assumed great importance, and has occupied the attention of your Council and members of the Society. The recent decision of a strong court of appeal in respect to the proper composition of mercurial ointment asserts and establishes this authority in unmistakable terms, and the result must have an important effect in future cases. I do not desire to discuss the whole question, and we shall have an opportunity this evening of hearing from Mr. A. H. Allen how the general question stands; but I desire to make a general remark on the subject. In the interests of the public health the purity of drugs is a matter of great importance, and it is the clear duty of pharmacists to uphold the official standards where they exist, since, if the authority of the standard is destroyed, unscrupulous persons will take the fullest advantage of the license to tamper with drugs, and will reduce their valuable and active constituents to a minimum. Free trade in drugs with no standards of purity is impossible, and would be disastrous.

I do not ignore the defects of the Pharmacopœia as it stands, but, in spite of such, it contains a large amount of valuable and authoritative material.

Among the results of the new Food and Drugs Acts has been the appointment of the Committees on Food Preservatives and the Standards to be adopted for Milk and Dairy Produce.

The Society, through myself and other members, has given evidence before both Committees, and has collected a quantity of information as to the experience of Public Analysts in various parts of the kingdom. We may hope the reports will not much longer be deferred, but the researches which the Preservatives Committee have instituted must be of necessity somewhat prolonged to establish the validity of their experimental work.

Already, however, some good can be traced from the inquiry, as I am convinced the use of preservatives is more restricted, and the excessive amounts only too frequently used formerly are now seldom met with.

In respect of milk standards, the Society may fairly congratulate themselves. Their standards were fixed over twenty years ago, and the question naturally arose whether the time had come to change them. The large experience of the application of the standard for solids-not-fat has shown it to be fixed at a fair and satisfactory limit, which, though not rigidly applicable to all circumstances, places and seasons, is substantially the correct standard limit if a single value must be adhered to. It does not cause injustice to honest dealers, and is adequate to check the addition of water save in limited quantity. No alteration, therefore, appears advisable. As to the Society's standard for fat, which its members have so long maintained through good and evil report, and all vicissitudes of season and weather, opinions are divided, and the standard which at one time was pronounced too severe, is now by some considered too lenient, and the Society was not unanimous in its recommendations, as some members desired to see a higher limit for fat than 3 per cent. There is no doubt that the limit could be raised for a part of the year, but there are objections to two standards, and I doubt if the higher figure could be insisted upon in cases of dispute. The decision of the Committee is not yet known, but we may expect to learn it very shortly.

One matter of great interest to analysts at present is the introduction of arsenic into various articles of food, owing to the use of arsenical sulphuric acid. Attention was called to a possibility of such contamination many years ago, but only recently have any cases been brought prominently under our notice. Last July a sample of effervescent phosphate of soda was found contaminated with arsenic, which led to the discovery that arsenic was very commonly present in phosphate preparations of various kinds, and arsenical contamination may occur in any preparations in the manufacture of which sulphuric acid is used. Now that attention has been so pointedly directed to the matter, we may expect that greater precautions in future will be taken by manufacturers to insure that only pure acid shall be used for the preparation of drugs, glucose, or any other articles of food. The responsibility rests clearly with the manufacturer. But under the present Food and Drugs Act glucose is manifestly included as an article used in the preparation of food, and samples can be taken by the inspectors on delivery at any brewery in the country by the request and with the consent of the purchaser.

There is one topic further I feel bound to mention. No former President, as far as I know, has had to ask the Society whether they desire to admit women to membership. Now that we have women Fellows of the Institute, there may be duly qualified candidates for admission to our Society, and the decision as to admission must rest in the end with members themselves. No formal nomination has yet been made, but the question as to such a step being permitted has already risen.

Before leaving the chair, I desire to thank, firstly, the Society for the honour they did me in electing me for their President, and next, the officers and all members of Council for their cordial support and kindly help in carrying on the Society's business, and specially those whose knowledge of its affairs goes back a long way in its history, and whose valuable co-operation has been in many important matters of great value to us collectively. And I welcome to the chair a successor distinguished by his public services to science and agriculture, and whose position there is typical of the widened interests of our Society, which are not limited strictly to the horizon of the Public Analyst, but embrace the whole field of analytical chemistry and those professionally associated with its applications and advancement.

ON THE ANALYSIS OF SAMARSKITE.

BY A. G. LEVY.

(*Read at the Meeting, December* 5, 1900.)

THE following account of the method employed in an analysis of samarskite recently made is published in the hope that it may be of use to other analysts.

In the choice of a method for attacking the mineral, preference was finally given to fusion with potassium bisulphate. In a preliminary attempt, in which the mineral was decomposed, according to Laurence Smith's method, by means of hydrofluoric acid, and the residue left after filtering this solution treated with a mixture of sulphuric and hydrochloric acids, some difficulty was experienced in obtaining a sharp separation of the acid from the basic oxides. In the potassium bisulphate method, on the other hand, the niobic and tantalic oxides are at once separated in the form of sulphates insoluble in water, together with the silica, and are only contaminated with a comparatively small quantity of other oxides, which can easily be removed by a second fusion with potassium bisulphate.

For two qualitative tests, however, portions of the mineral were decomposed with HF and HCl, and the solution obtained examined for ferrous iron and titanium, by adding potassium ferricyanide solution to one portion and a piece of zinc to the other; in neither case was a coloration observed; hence all the iron was in the ferric state, and titanium was absent.

For the complete analysis, 1 gramme of the finely powdered mineral was fused with about 6 grammes of potassium bisulphate, in a platinum crucible, until it appeared to be thoroughly decomposed. The cold melt was digested with warm water until completely disintegrated, the residue filtered off, washed, strongly ignited, and weighed as crude oxides of silicon, niobium, and tantalum. Its further treatment is given later on.

To the solution containing the soluble sulphates some hydrochloric acid was added, and then excess of ammonia, the operation being performed in a flask so as to exclude CO_2 as far as possible, in order to precipitate uranium completely. Notwithstanding this precaution, some uranium was found in the filtrate from this precipitate, as well as in most of the others subsequently obtained from precipitates containing uranium, coming down on long standing or boiling. The excess of ammonia was removed from the filtrate by evaporation, and the additional precipitate of alumina collected. Lime and magnesia were estimated as usual in the filtrate, neither zinc nor manganese being present.

The two ammonia precipitates were dissolved in hydrochloric acid, and alumina separated by caustic soda; the rare earths were then thrown down as oxalates, and converted to oxides by ignition; and, in the filtrate, iron and uranium separated by means of ammonium sulphide and carbonate. As the ferric oxide so obtained contained some uranium, the separation had to be repeated. Uranium was finally precipitated by ammonia, and the precipitate ignited and weighed first as U_3O_8 and then as UO_2, the weights agreeing very nearly with each other.

To purify the crude mixed oxides of silicon, niobium and tantalum, from any iron, etc., they might still contain, they were reduced to a fine powder, and 0·2000 gramme of this taken for a second fusion with bisulphate, the melt being treated as before; the small quantities of iron, aluminium and uranium found in solution, were estimated as above. As the insoluble residue from the melt, containing silicon, niobium and tantalum, was, after ignition, perfectly white when cold, the original oxides being yellowish-brown in colour, it was considered as being free from iron and uranium.

An attempt was then made to separate the niobic and tantalic oxides as follows: About 3 grammes of potassium hydrogen fluoride, obtained from pure $KHCO_3$ and pure HF, were fused in a platinum crucible, allowed to cool, and the mixed oxides added, after which the salt was again cautiously heated to fusion. A brisk reaction took place, the oxides dissolving readily. The melt was then allowed to cool, dissolved in water, and the solution evaporated to a very low bulk and allowed to crystallize. The crystals which contain tantalum as K_2TaF_7 were filtered off from the solution in which niobium is present as K_2NbOF_5 and washed repeatedly with very small quantities of water. Both solution and crystals were then heated with excess of H_2SO_4 till all HF had been expelled, the residue was extracted with hot water and filtered off, niobic and tantalic sulphuric acids being left behind; but by this procedure only about one-third of the niobium in solution was obtained, as mere evaporation with H_2SO_4 apparently does not render it insoluble; on taking down the filtrate to actual dryness, the remainder was found as a residue insoluble in water.

As the silica is of course volatilized during the fusion with HF,KF, it was estimated separately, both on another portion (0·1 gramme) of the crude oxides and on 0·5 gramme of the ignited mineral, by treatment with HF and H_2SO_4, and igniting to constant weight. It amounted to 2·86 per cent. on the ore, or 5·03 per cent. on the crude oxides. On now adding this calculated quantity of silica to the quantities of iron, aluminium, uranium, niobic and tantalic oxides found, a total of only 0·1855 gramme was obtained, instead of the 0·2000 gramme taken.

To find the cause of this loss, some experiments were made with commercial niobic and tantalic oxides. On making a mixture of these, and treating them by the process described above, a similar loss was experienced. The behaviour of Ta_2O_5 and Nb_2O_5 on treatment with HF, and with HF and H_2SO_4, followed by evaporation to dryness, and ignition of the residue, was then examined, and it was found that neither niobic nor tantalic oxide volatilizes when treated with HF and H_2SO_4, and that niobic oxide does not form volatile compounds even when treated with HF alone, but that tantalic oxide, by the action of HF, loses in weight each time the operation is repeated, resembling titanium in this respect. In one experiment a bluish sublimate was actually noticed on the sides of the crucible. It appeared to be difficult to expel the fluorine completely from the residue left after treatment with HF, by mere ignition in air, but this could easily be accomplished by heating first with H_2SO_4 and then igniting.

These experiments seemed to show that the loss noticed in the fusion method was due to a portion of the tantalum volatilizing as a fluorine compound during the heating. Fusion with HF,KF had been resorted to under the impression derived from Fresenius ("Qual. Analysis"), that the ignited oxides were insoluble in aqueous HF; this statement can be traced to Marignac (*Ann. Chim. Phys.*, 1866, iv. 8, 16); but as during the above experiments it had been noticed that they dissolve readily in the warm acid, the following method was now tried: Another portion of the crude oxides was fused with $KHSO_4$ for purification from iron, etc.; from the residue obtained on treating the melt with water SiO_2 was removed by means of HF and H_2SO_4; the ignited oxides of Nb and Ta were dissolved in HF, and this liquid poured into a solution of HF,KF, when a crystalline precipitate was formed which dissolved on heating. In order to convert the double fluoride of potassium and niobium to the oxyfluoride, the excess of HF was removed by evaporating the solution to dryness on the water-bath, the residue taken up in a little water, and this solution allowed to crystallize. Solution and crystals were then treated with H_2SO_4, as in the fusion method. The weights of Nb_2O_5 and Ta_2O_5 obtained agreed almost exactly with the quantity of mixed oxides taken. As the separation is at best only a fractional one, it was repeated on both the niobic and tantalic oxides obtained, small quantities of insoluble and soluble salts respectively being found, which were added to the main quantities. The weights obtained in these two separations again showed close agreement with the weights taken, and the method seems to be reliable.

A rough separation of the rare earths into ceria and yttria groups was next made. For this purpose the oxides obtained by igniting the oxalate precipitate, which were of a light brown colour, were treated first with nitric acid, and then, as this failed to effect complete solution, after evaporating off the nitric acid, with hydrochloric acid and a little potassium iodide to dissolve any cerium dioxide. A small residue which still refused to dissolve was filtered off, ignited, and weighed; it was not identified, but certainly was not CeO_2, as was at first suspected. The solution containing the bulk of the earths was evaporated to dryness, and the residue treated with a little water, and then with a saturated solution of potassium sulphate; after standing for two days the crystalline precipitate was filtered off, and washed with K_2SO_4 solution; to the filtrate hydrochloric acid and then ammonia were added; the resulting precipitate after washing was dissolved in hydrochloric acid, again thrown down by ammonia, washed, ignited, and weighed as "oxides belonging to the yttria groups." It was brown in colour.

The precipitate of double sulphates was dissolved in hydrochloric acid, precipitated by ammonia, and the hydroxides obtained washed, ignited, and weighed as "oxides belonging to the ceria groups." They were white in colour, and dissolved completely in strong hydrochloric acid with evolution of chlorine, although no cerium could be detected either by the PbO_2 and HNO_3, or by the hypobromite tests. In a larger quantity of oxides obtained by decomposing 10 grammes of the mineral cerium was also absent. No further separation of the rare earths was attempted. A spectroscopic examination revealed the presence of didymium, but no absorption bands due to samarium could be seen.

The loss on ignition of the mineral was determined both at a dull red heat and at a bright red heat. The second value was a little greater than the first. The samarskite was not examined for alkalies.

The composition of the samarskite, calculated from the analytical data, is given below:

	Per Cent.
Silica	2·86
Niobic oxide	31·52
Tantalic oxide	16·63
Ferric oxide	10·68
Aluminium oxide	3·07
Uranium trioxide	12·13
Calcium oxide	3·34
Magnesium oxide	0·10
"Cerium" oxides	1·65
"Yttrium" oxides	14·32
"Insoluble" oxides	0·79
Loss on ignition	2·48
Total	99·57

In conclusion, I should like to state that the analysis was carried out in the laboratory of Mr. Bertram Blount, to whom I am also indebted for much valuable help.

DISCUSSION.

Mr. BLOUNT said that this paper was especially interesting because of the fact that the separation of some of the bodies referred to was not easily achieved by the methods to be found in the text-books. The work which had to be done was of a pioneer character. It was necessary first of all to obtain reasonably pure samples of the various oxides and so forth, and, by systematically examining them, to get their reactions into one's mind. Then the statements of the text-books had to be corrected, and finally the analysis made; so that an analysis represented a piece of work really much greater than appeared from the mere figures detailing the chemical compounds of which a particular mineral might be made up. It seemed interesting that there should be so much difference in the behaviour of tantalic and niobic acids. The volatility of tantalic acid under the conditions named should be regarded in any exact analysis of a mineral of this class. There seemed in the case of these substances to be a range of volatility, from the absolute volatility of silica treated with a mixture of sulphuric and hydrofluoric acids, to the limited volatility of tantalic acid and titanic acid, which disappeared with hydrofluoric acid, but were retained with sulphuric acid, and the absolute non-volatility of, say, stannic oxide. With regard to the occurrence of what Mr. Levy had termed "insoluble rare earth oxides," he believed that the efforts which had been made to identify these had resulted in nothing very definite, because the quantity of the oxides was so small. There was no doubt, however, that they belonged to the group to which they were assigned, although they were insoluble.

Mr. HEHNER said that his assistant, Mr. P. W. Skertchly, had found that, contrary to the text-book statement that tungstic oxide could be readily separated from stannic oxide by volatilization of the latter on the addition of ammonium chloride, the tungstic oxide also volatilized under such circumstances, its volatilization increasing with the quantity of ammonium chloride added. There was certainly a rough distinction, but this was not sharp enough for analytical purposes.

Dr. RIDEAL said that he was able to confirm Mr. Hehner's remark as to the volatility of tungstic oxide in presence of ammonium chloride. Perhaps he might suggest that, in investigating a mineral containing rare metals of the uranium class, one ought to examine the material spectroscopically, and also ascertain whether the Becquerel rays showed the presence of polonium or radium.

Mr. C. T. TYRER said that he had had some experience in the examination of pitch-blende, gadolinite, and other minerals of a similar character; and he had come to the conclusion that the success of either their analytical or their technical treatment depended entirely upon the preliminary treatment to which they were subjected. Some years previously there had been discovered what was thought to be a new metal, which turned out to be a mixture of niobium or niobic oxide and tungstic acid, which was very difficult to separate.

Mr. LEVY said that at the time when this analysis was made the niobic and tantalic oxides, and not the uranium, had formed the subject of chief importance, so that the consideration of the uranium had really to a great extent been neglected.

THE OCCURRENCE OF BARIUM IN THE SPRING WATER OF BOSTON SPA.

BY PERCY A. E. RICHARDS, F.I.C.

(Read at the Meeting, December 5, 1900.)

THE presence of any considerable amount of a soluble barium salt in a natural water is of such rare occurrence that the present paper may prove of interest to the Society.

The village of Boston Spa, with its neighbour Thorp Arch, lies on the banks of the river Wharfe, about ten miles from Harrogate. About 100 years ago the place enjoyed quite a reputation for the medicinal properties of its water, then thought to be chalybeate in character.

With the rise of Harrogate the popularity of this Spa declined, until at the present day only a comparatively few people know the spring, and these are mainly visitors to the village during the summer months.

In "York and East Riding," vol. i., 1855, there is a very old analysis of the water, stated to have been made by Dr. Adam Humber, of Leeds, in the year 1784. The figures obtained are given below:

WATER OF BOSTON SPA.

Contents of One Wine Gallon.

	Grains.
Muriate of soda...	562·00
Muriate of lime ...	12·25
Muriate of magnesia	7·25
Carbonate of iron	1·75
Silica ...	0·75
	584·00
Gases :	Cubic Inches.
Carbonic acid	10·56
Azotic gas ...	6·00

A short time ago I had the opportunity of making an analysis of the water with a view of ascertaining whether there was an important amount of iron present or not. For this purpose I visited the spring, and took some samples myself.

When first pumped to the surface the water was perfectly bright, clear, and sparkling, with a slightly bitter taste. After standing for about an hour, even in a stoppered bottle, it became opalescent, and finally deposited a small precipitate, which consisted mainly of oxide of iron. It was found upon examination that comparatively very little of this metal was present, but that the water contained a distinct amount of the unsuspected element barium.

The figures obtained upon analysis are given below:

WATER OF BOSTON SPA.

Results expressed in Parts per 100,000.

Total solid constituents (dried at 100° C.)	1271·00
Total solid loss on ignition	251·00
Saline residue ...	1020·00
Total alkalinity (calculated as $CaCO_3$)	25·10
Alkalinity after boiling (calculated at $CaCO_3$)	1·50
Ferric oxide (Fe_2O_3)	1·01
Calcium oxide (CaO)	49·64
Barium oxide (BaO)	30·16
Magnesium oxide (MgO) ...	24·06
Potassium oxide (K_2O)	1·10

Sodium oxide (Na_2O)	555·11
Silica (SiO_2)	1·21
Free and saline ammonia (NH_3)	0·22
Albuminoid ammonia	0·008
Sulphuric anhydride	nil
Phosphoric anhydride	nil
Chlorine	735·22
Bromine	0·60
Iodine	nil

Specific gravity at 15° C.: 1010.
Appearance in 2-foot tube: brownish green.

Dissolved Gases:	Cub. In. per Gal.
Nitrogen	3·27
Carbonic acid	17·87

The water apparently varies in composition to a certain extent, and this is shown by the following figures, taken from the results of another analysis made six months after the first:

Parts per 100,000.

Barium oxide	25·81
Calcium oxide	46·08
Magnesium oxide	20·78
Sodium oxide	457·83
Chlorine	614·28
Total solid constituents	1050·00

The following probably represents the actual salts present in the water calculated on the first analysis:

Results expressed in Parts per 100,000.

Barium chloride	41·00
Calcium chloride	69·94
Magnesium chloride	56·91
Sodium chloride	1047·59
Potassium chloride	1·71
Sodium bromide	0·77
Calcium carbonate	25·10
Ferrous carbonate	1·50
Silica	1·21

The water was tested spectroscopically for strontium, but failed to give any evidence of the presence of that element.

The soil of the neighbourhood consists of sand, clay, and magnesia limestone, but the rocks are chiefly calcareous. Barium has been found on one or two occasions previously in natural waters both in England and Wales. In the water of the Llangammarch Well it occurs, according to the analyses of Dupré and S. A. Vasey, to the extent of about 9·45 parts per 100,000. Again, in an interesting paper read before this Society by J. White in January, 1899, analyses were given of waters drawn from deep-well borings at Ilkeston, Derbyshire, containing as much as 38·5 to 40·7 parts per 100,000 of barium chloride.

These waters have all very much the same character, containing considerable amounts of calcium and magnesium chlorides, together with a very large proportion of sodium chloride.

A curious point about the Boston Spa spring is that the water has been taken internally, under the guise of a chalybeate water, for more than 100 years without the presence of the barium being in the least suspected.

DISCUSSION.

Mr. John WHITE said that the chief points of difference between the Boston Spa water and the samples from Ilkeston, which he had examined, were that, while the former contained more barium oxide than the latter, it only contained about half the quantity of solid matter and half the quantity of chlorides as compared with the amounts present in the Ilkeston waters. In the case of the Ilkeston waters he (Mr. White) had come to the conclusion that the barium existed as carbonate, in one instance, and as chloride in the others. The author had not referred to carbonate, but had calculated the barium present as chloride, with which view he agreed.

Mr. FAIRLEY said that most of the Harrogate waters contained barium, generally in very small quantity, but as a rule sufficient to prevent the finding of any sulphuric acid in solution. The quantity of barium being small, it went down with the silica as barium sulphate, and unless the silica was carefully examined the barium might be overlooked. In the detection of strontium it was necessary to apply a process of careful separation and concentration to the calcium precipitate; and even then very small a quantity of strontia was easily missed.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

Note on the Reichert Value of Butter and other Fats. A. Reychler. (*Bull. Soc. Chim.*, 1901, xxv., 142-144.)—The author suggests a slight extension of the Reichert-Meissl process so as to include not only the empirical proportion of soluble fatty acids, but also the amount of volatile insoluble acids obtained under the same conditions.

Instead of filtering the distillate, he adds 50 c.c. of alcohol, and if necessary a little ether, and titrates the solution in the usual way. The ratio thus obtained between the value for the total volatile acids and for the soluble volatile acids varies greatly in different fats, the difference being most marked between butter and cocoanut oil, as is shown in the following table :

	I. Total Volatile Acids.	II. Soluble Volatile Acids.	III. Ratio of II. and I.
1. Butter	32·4	29·36	0·91
2. Cocoa-nut oil	22·7	7·4	0·33
3. Mixture: 79·4 % butter 20·6 % cocoa-nut oil ...	31·4	24·4	0·78
4. Butter	32·0	28·6	0·89
5. Cocoa-nut oil	22·2	7·2	0·32
6. Mixture I.: 75 % oleo-margarine 25 % neutral lard	1·50	0·83	0·55
7. Mixture II.: 50 % of Mixture I. 50 % cocoa-nut oil	12·45	5·6	0·45
8. Mixture III.: 19·75 % of Mixture II. 80·25 % of butter	28·6	24·3	0·85

C. A. M.

Detection of Saccharin in Wine or Beer in the Absence of Salicylic Acid. F. Wirthle. (*Chem. Zeit.*, 1900, xxiv., 1035.)—This is an adaptation of C. Schmitt's process, based on the conversion of saccharin into salicylic acid by heating it with sodium hydroxide. One hundred c.c. of wine (200 if necessary) are concentrated to 20 c.c., rinsed into a separating funnel with a little caustic soda, strongly acidified with sulphuric acid, and extracted three times with 50 c.c. of ether. The latter is filtered, treated with 10 c.c. of 0·5 per cent. soda, and the solvent distilled off. The residue is brought into a small basin with water and a little soda, 1 gramme of solid sodium hydroxide introduced, and the whole heated slowly to 215° C., maintaining the temperature between 210° and 220° for fifteen minutes. The cold melt is taken up in warm water, cautiously acidified with sulphuric acid, and extracted with a mixture of ether and petroleum spirit. The solvents are removed, a few drops of water added, and a very weak solution of ferric chloride dropped in. One milligramme of saccharin in the wine gives a brilliant violet colour; whereas wines free from saccharin, even red wines with much tannin, yield at most a dirty orange. It is advisable to carry out a blank experiment with 1 milligramme of saccharin dissolved in spirit, mixed with 1 c.c. of caustic soda solution, and heated with 1 gramme of alkali as before.

Beer requires a preliminary treatment with a few c.c. of saturated copper acetate solution followed by sodium phosphate; the filtrate is then extracted with ether as above. Decoctions of hops respond to the test, showing the characteristic violet tint with 1 milligramme of saccharin, while in its absence they give at the utmost a faint rose colour.

[*Cf.* Hasterlik, ANALYST, 1899, xxiv., 152.] F. H. L.

A Source of Error in Testing for Salicylic Acid in Portuguese Wines. A. J. Ferreira da Silva. (*Bull. Soc. Chim.*, 1900, xxiii., 795, 796.)—The Customs authorities of Brazil have recently condemned several Portuguese wines on the ground of their containing traces of salicylic acid. The author, however, asserts that salicylic acid is not added to wine in Portugal, and attributes the error of the Brazilian chemists to their having used the method of Pellet-Grobert and Baudrimont ("Dict. des Altérations et Falsifications," 1897, p. 253), in which 200 c.c. of the wine are extracted with ether. He has found that certain Portuguese wines contain a small quantity of a substance having some resemblance to salicylic acid, and of which sufficient can be extracted with ether to give a rose coloration with ferric chloride.

By using the official German method, however, in which 50 c.c. (not more) of the wine are acidulated and shaken with a mixture of equal volumes of ether and petroleum spirit, he has never obtained this coloration.

An analogous phenomenon was observed in certain German and Austrian wines by Medicus (*Berichte, 9th Versammlung bayerischer Vertreter Angew. Chem.*, 1890, pp. 42-48), who concluded from direct experiment that the substance resembling salicylic acid was derived from the stalks of the grapes. C. A. M.

Adulteration of Roasted Coffee by Treatment with Borax and Water. E. Bertarelli. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1900, iii., 681-683.)—The author calls attention to the adulteration of roasted coffee-beans by the practice of pouring over them a boiling aqueous solution of borax. The solution is readily absorbed by the beans, causing an increase of about 12 per cent. in their weight, and renders them glossy in appearance, without at all impairing their original hardness. Borax should be looked for in cases where 4 per cent. or more of water is found. Whereas genuine roasted coffee does not usually contain more than about 3 per cent. of water, the author has found upwards of 10 per cent. in roasted beans which have been treated in the way described. H. H. B. S.

Artificial Colouring of Oranges. Pum and K. Micko. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1900, iii., 729-735.)—The authors challenge the accuracy of a statement made by Hotter, to the effect that oranges were being exposed for sale as "blood-oranges" which were in reality ordinary yellow oranges which had been injected with an aniline or a vegetable dye. They not only consider Hotter's methods of detection (mainly spectroscopic) to be inconclusive, but they declare it to be a practical impossibility to so inject a coloured fluid into an orange as to produce throughout the whole a fair resemblance to the natural fruit. H. H. B. S.

Commercial Bismuth Subnitrate. F. A. Upsher Smith. (*Pharm. Journ.*, 1900, 692.)—The author has examined four specimens of bismuth subnitrate o English manufacture. He found that the form, density, and composition varied with the conditions of precipitation. The salt does not correspond with the British Pharmacopœia formula, $BiO \cdot NO_3 \cdot H_2O$, which requires 76·367 per cent. Bi_2O_3, 17·724 per cent. N_2O_5, and 5·908 per cent. H_2O. The samples examined contained 79 to 81 per cent. Bi_2O_3, 17·5 to 19·5 per cent. N_2O_5, and 2 to 3 per cent. H_2O; these results agree fairly with those given by other observers.

The author recommends that the determination of bismuth should be made by ignition to oxide, if inorganic impurities are present after precipitation with ammonium carbonate, instead of by weighing as sulphide. He recommends Kebler's indirect process for determining the combined acid: 2 grammes of the sample, mixed with 5 c.c. of water, are boiled for half an hour with 10 c.c. N potassium hydroxide solution; the precipitate is filtered off, washed, and the filtrate titrated with $\frac{N}{10}$ hydrochloric acid, phenolphthalein being used as indicator. Methods in which potassium hydroxide acts on the salt in the cold give results 3 to 4 per cent. too low. The water is determined by heating the salt at 120° for several hours. A. G. L.

ORGANIC ANALYSIS.

The Detection of Methyl Alcohol in Mixtures. S. P. Mulliken and Heywood Scudder. (*Amer. Chem. Journ.*, xxiv., 444.)—The authors have examined the modification of E. Jandrier (*Ann. Chim. Anal.*, App., 4, 156), who recommended the substitution of gallic acid for resorcinol, and have further improved their test for methyl alcohol (*Amer. Chem. Journ.*, xxi., 266). They found that the gallic acid reaction is so delicate that it is useless, since the reaction is obtained with the oxidation products of a large number of organic substances, such as ethyl alcohol, glycerol, and acetic acid, owing to the formation of traces of formic aldehyde when these substances are oxidized. On the other hand, methyl esters and ethers are the only common organic compounds besides methyl alcohol which give the reaction with resorcinol.

If the test is applied to a mixture of unknown composition, only the portion distilling between 50° and 100° C., and soluble in 2 or 3 volumes of water, should be used. From phenols and acids, methyl alcohol can be separated by distilling the alkaline solution, from bases by distilling the acidified liquid. A blank experiment should also be made by pouring 2 c.c. of the unoxidized solution, to which 1 drop of a 0·5 per cent. resorcinol solution has been added, on to concentrated sulphuric acid, so as to form a layer. If a coloration or precipitate is obtained, and the solution gives the usual aldehyde reactions, formic aldehyde is removed by heating 12 c.c., diluted, if necessary, so as to contain 75 per cent. of water, with 3 grammes resorcinol and 1 c.c. sulphuric acid, in a stoppered bottle, for two hours at 70° to 80° C. The solution is then diluted to 50 c.c., 5 c.c. distilled off, and tested as usual. Allen and Chattaway's method (ANALYST, xvi., 102) may also be used. This preliminary treatment is seldom necessary in the case of fermented and distilled liquors.

If the solution contains ethyl alcohol, the acetaldehyde formed on oxidation interferes with the delicacy of the test. It can be removed as follows: Two c.c. of the solution, diluted, if necessary, to 6 c.c., are oxidized with the copper spiral. The test-tube is then closed with a rubber stopper, through one hole of which a tube, drawn out to a very fine capillary almost touching the bottom of the test-tube, is inserted; the upper end of this tube can be closed by a screw clamp fitted to a rubber tube; by a second tube inserted in the stopper the test-tube is connected with a good suction-pump. The tube is kept at 25° to 30°, and exhausted, the capillary serving to prevent bumping. Acetaldehyde evaporates readily, whilst formic aldehyde remains behind. When one half of the liquid has evaporated, 1 drop of the 0·5 per cent. resorcinol solution is added, and it is poured on to sulphuric acid in another test-tube. After standing for three minutes, the test-tube is agitated very gently for about one minute. The separation of rose-red voluminous flocks indicates methyl alcohol, but a mere coloration, or flocks of a different colour, should not be accepted as proof of its presence. The test shows the presence of 1 part of methyl alcohol in 2,000 of the diluted solution, and is satisfactory when the proportion of ethyl alcohol does not exceed 100 to 3. It is also given by secondary and butyl alcohols and by methyl ethers and esters. A. G. L.

Estimation of Ash in Commercial Glycerin. C. Ferrier. (*Monit. Scient.*, 1900 [4], xiv., 808; through *Chem. Zeit. Rep.*, 1900, 364.)—Ten grammes of the sample are cautiously evaporated in platinum, and the tarry residue is carbonized. It is broken up into powder, 5 or 6 c.c. of water added, the whole allowed to soak for a few minutes, and the liquid removed with a narrow-ended pipette. The treatment with water is repeated; the coke is dried and ignited. When the carbon has been burnt off and the basin is cold, the aqueous extract is returned to the vessel, the mixture thoroughly dried, and ignited again for a few seconds. By this method of manipulation fusion or volatilization of the alkali metal salts is avoided, and the results are constant to within 0·01 per cent. F. H. L.

A Delicate Test for Dextrose and Aldehydes. E. Riegler. (*Wiener med. Bl.*, 1900, xxiii., 785; through *Chem. Zeit. Rep.*, 1900, 383.)—One c.c. of the solution to be examined, about 0·5 gramme of crystallized sodium acetate, and 2 c.c. of a fresh 2 per cent. solution of phenylhydrazine hydrochloride are boiled together, 10 c.c. of 10 per cent. sodium hydroxide are added immediately, and the whole shaken. Presence of dextrose causes the formation of a fine rose colour, which appears at once or in fifteen or thirty minutes, according to the proportion of sugar in the liquid. Any colour developing after thirty minutes is neglected. The limit of delicacy of the test is 0·005 per cent. Uric acid, creatinine, and albumin do not interfere; therefore the reaction should be useful in analysing urine. It can also be employed to detect formaldehyde and other aldehydes. F. H. L.

The Estimation of Uric Acid. A. Bellocq. (*Journ. Pharm. Chim.*, 1900, xii., 103, 104.)—To 250 c.c. of the urine under examination is added an excess of sodium hydroxide solution, the precipitate allowed to subside, and the supernatant liquid decanted, shaken with asbestos powder and filtered. To 200 c.c. of the filtrate are added 20 c.c. (or more if required) of a reagent consisting of—zinc sulphate solution (1 : 3), 30 c.c.; sodium hydroxide solution, 30 c.c.; and sodium carbonate solution (saturated), 40 c.c.

The voluminous precipitate is collected on a filter and drained. It is next detached from the paper and dried in a porcelain crucible, into which are then introduced 2 or 3 c.c. of hydrochloric acid saturated with pure uric acid. The crucible is allowed to float on the surface of cold water, and the uric acid crystals which deposit are collected, washed with 10 c.c. of alcohol, dried and weighed.

C. A. M.

Notes on the Estimation of Uric Acid and Purine Bases in Blood, etc. W. His and W. Hagen. (*Zeits. physiol. Chem.*, 1900, xxx., 350; through *Chem. Zeit. Rep.*, 1900, 319.)—Finding all the processes hitherto described unsatisfactory, the authors have submitted the whole question of determining uric acid and purine bases in animal organs to a fresh investigation, and they record the following observations. When albumoses are present in a large proportion in a guanine solution, they hinder the formation of the silver precipitate; if the proportion is small, they are partly thrown down with the guanine. The amount of albumose-nitrogen in the precipitate increases with the quantity of albumoses in the solution, and also with the ratio they bear to the guanine. If the nitrogen of the silver precipitate is estimated, even a small proportion of albumose (0·006 per cent.) in the liquid causes considerable errors (15 per cent.). Even 0·05 per cent. of albumose may prevent the formation of the silver salt; whether a precipitate is produced or not depends on the mass of the solution, on the ratio of the albumoses and the guanine to the volume of the liquid, and on their ratio to one another. Ammonium sulphate assists the formation of a precipitate in a liquid containing albumoses. In a cold saturated solution of the sulphate, or in a 10 per cent. solution of trichloracetic acid, the basic nitrogen can be determined by precipitation with ammoniacal silver; in a solution of zinc sulphate this is not possible. The silver compound of guanine recovered from a liquid containing albumoses carries some of the latter down with it; but it may be purified by one to three reprecipitations. Addition of ammonium sulphate or trichloracetic acid determines a silver precipitate in solutions rich in albumoses; but the deposit contains albumoses. Attempts to remove the albumoses before treatment with silver by means of ammonium sulphate and iron alum cause noteworthy loss of the bases; trichloracetic acid is incomplete in its action, and the precipitate still contains albumoses; zinc sulphate is satisfactory, but the excess must be removed by sulphuretted hydrogen; ammonium sulphate in weak sulphuric acid solution is also satisfactory, provided the liquid is saturated in the cold and is allowed to rest for twenty-four hours. In a concentrated solution of ammonium sulphate, precipitation with silver is imperfect; in a 25 per cent. solution it is complete. When applied to the extracts of animal organs, estimation of basic nitrogen by the ammonium sulphate process causes considerable errors, too little nitrogen being found. Inasmuch as the method is satisfactory with liquids which contain only albumoses, it must be assumed that the organs contain substances beside albumoses which prevent the formation of the silver guanine compound; possibly these are the nuclein acids, whose purine salts are not wholly precipitable with silver. On one occasion the lead acetate method gave a good result; on two others the deficit was large. It is therefore evident that there is no general process available for estimating the bases. Precipitation of uric acid as a silver-magnesium compound is not hindered by a small quantity of albumoses. For its determination in organs, Stadthagen's plan of extracting with 0·5 per cent. sulphuric acid is good; properly conducted, it gives a yield of 90 to 97 per cent. The ammonium sulphate and lead acetate method is not suited to the determination of uric acid; therefore the sulphuric acid extract of animal organs should be divided into two portions for estimating uric acid and the bases. F. H. L.

A Comparison of the Amounts of Nitrogen precipitated by Various Proteid Reagents. **H. Schjerning.** (*Zeit. anal. Chem.*, 1900, xxxix., 545-566.)—The author has determined the percentage of nitrogen precipitated by his six proteid reagents (ANALYST, xxiii., 104 and 326) and by other reagents from different non-proteid nitrogenous substances, and gives his results in the following table:

Substance.	N in Substance, corresponding to c.c. of N/10 Acid.	Tin Chloride.	Mercuric Chloride.	Lead Acetate.	Ferric Acetate.	Uranium Acetate.		Magnesium Sulphate.	Bromine-water.	Tannin.	Stutzer's Reagent.	Phospho-tungstic Acid.	
						Without P_2O_5.	With P_2O_5.					Cold.	Boiling.
Ammonium acetate ...	24·4	—	12·3	—	—	—	—	—	—	—	—	×	×
Neurine	3·9	—	✱	✱	✱	—	—	—	—	15·4	✱	69·2	71·8
Betaine HCl	6·8	—	—	✱	✱	—	—	—	—	—	—	38·2	27·9
Guanine	4·0	—	—	—	✱	—	✱	—	—	—	47·5	45·0	—
Alloxan	9·1	26·4	—	3·3	24·2	—	—	—	—	—	31·9	✱	—
Allantoïn	13·8	—	—	—	✱	—	—	✱	—	—	2·9	—	—
Kreatine	8·0	✱	—	2·5	—	—	—	—	—	—	2·5	5·0	—
Asparagin	15·3	—	—	—	✱	—	4·5	—	—	—	✱	2·5	—
Leucin	6·1	—	—	—	—	—	—	—	—	—	✱	—	—
Arginin	12·5	—	—	—	—	—	18·4	—	—	—	4·0	79·2	✱
Uric acid	1·6	—	—	—	—	—	—	—	—	—	31·3	—	—
Hippuric acid	3·2	—	—	—	✱	—	—	25·0	—	—	—	—	—
Tyrosin	2·6	—	—	—	—	—	—	—	×	—	✱	—	—
Piperazine	3·8	—	94·7	✱	✱	44·7	71·0	—	68·4	✱	✱	89·5	36·8
Caffeine	15·9	—	4·4	—	1·3	✱	—	94·3	×	×	—	91·2	93·7
Theobromine	8·8	—	—	—	—	—	—	5·7	—	—	—	×	—
Quinine	5·7	—	×	3·5	3·5	—	—	52·6	61·4	×	3·5	96·5	98·2
Morphine	3·8	—	—	—	✱	—	—	—	5·3	—	—	100·0	84·2
Brucine	9·6	—	—	—	—	—	2·1	25·0	11·4	×	—	101·0	?
Amygdalin	8·1	—	—	✱	✱	—	—	19·8	—	—	—	—	—
Solanine	1·5	—	—	—	—	—	—	66·7	×	—	—	—	—

In this table and the subsequent table × denotes that the precipitate produced was so finely divided that it could not be collected. The sign ✱ signifies that the trifling precipitate formed was within the limits of experimental error. In the cases marked ? there was a considerable precipitate which was lost. In the other cases there was no precipitate.

From these results the author concludes that tin chloride, mercuric chloride, lead acetate, and uranium acetate must be regarded as the most reliable precipitants for proteids in the presence of non-proteid nitrogenous substances.

The precipitation of ammonium from its salts by mercuric chloride only occurs when the ammonium is in combination with an organic acid, and when equivalent quantities of chlorides are not present. Thus, no precipitate is formed on adding an excess of mercuric chloride to a solution containing equivalent quantities of ammonium acetate and ammonium chloride.

Magnesium sulphate added to saturation precipitates numerous non-proteid nitrogenous substances, but the author remarks that these will not usually occur in proteid estimations; when they do so, the precipitations must be made exclusively with tin chloride, mercuric chloride or lead acetate, ferric acetate, and uranium acetate.

He objects to bromine-water as a reagent as used by Allen and Searle (ANALYST, xxii., 258), on the ground that the precipitates cannot be prevented from passing through the filter, even when a little magnesium sulphate has been added.

Similar comparative determinations were made with different proteid solutions, with the following results, showing the percentages of the total nitrogen precipitated :

Substance.	Tin Chloride.	Mercuric Chloride.	Lead Acetate.	Ferric Acetate.	Uranium Acetate.		Laszczynski's Method.	Bromine-water.	Tannin.	Stutzer's Reagent.	Phospho-tungstic Acid.	
					Without P_2O_5.	With P_2O_5.					Cold.	Boiling.
Separated milk	81·9	90·5	89·6	90·5	91·4	91·4	90·5	76·7	91·4	?	90·5	91·4
Blood serum	?	97·2	×	98·2	99·1	99·1	98·2	88·0	100·0	97·2	98·2	100·0
Liebig's meat extract	6·6	15·8	14·5	23·7	32·9	42·1	13·2	×	21·1	32·9	56·6	26·3
Witte's peptone	2·9	30·2	×	57·0	53·5	82·0	49·4	32·0	70·3	59·3	88·9	83·7
Yeast wash	34·2	41·7	39·2	52·5	60·8	65·0	×	11·8	33·3	46·7	?	13·3
Beer	5·5	17·3	16·6	33·1	42·8	44·1	20·7	8·3	29·0	38·6	44·1	27·6

Laszczynski's method (*Zeit. gesammt. Brauwesen*, xxii., 124 and 141) consists in heating the proteid solution for an hour at a pressure of 1·5 atmosphere, collecting the coagulum on a filter, and washing it two or three times with warm water. In the author's opinion this only causes the coagulation of all the true albumins, and he considers that the results obtained with blood serum and milk show that it is not generally applicable.

As regards these bromine precipitations, he asserts that the results obtained with milk and blood serum, both of which contain practically only proteid nitrogen, show that bromine cannot be used as a general method of separating proteid and non-proteid constituents.

Phosphotungstic acid, like uranium acetate, precipitates all proteids (ANALYST, xxiii., 186), but in many cases it cannot be used owing to the presence of certain non-proteid nitrogenous substances. Attempts to obviate this by making the precipitation from a boiling solution were only partially successful.

Tannin, according to the author, does not precipitate albumoses (propeptones) or true peptones, and another objection to it is that it precipitates many non-proteid nitrogenous compounds.

As regard's Stutzer's reagent, he points out that many amido-compounds are precipitated by it, as copper salts ; and that, on the other hand, it does not quantitatively precipitate true peptones.

In conclusion, he remarks with reference to proteid determinations in general, that when the solution contains a relatively large quantity of mineral salts of light metals, a quantitative estimation of the different proteid groups cannot be made with certainty, since the resulting compounds react reciprocally with these salts, forming soluble proteid compounds of the light metals. The only exceptions to this rule are the precipitations (" salting out ") with magnesium sulphate, with tin chloride, and probably also with Stutzer's reagent and with tannin. C. A. M.

Behaviour of Albuminous Substances towards Alkaloid Reagents. Estimation of Combined Hydrochloric Acid in the Stomach. O. Cohnheim and H. Krieger. (*Zeits. Biol.*, 1900, xl., 95 ; through *Chem. Zeit. Rep.*, 1900, 262.)—Albumin, albumoses, and peptones in aqueous solution behave as bases, combining with acids to form "salts," of which the hydrochlorides are of interest. The hydrochlorides of albumoses are acid to litmus, phenolphthalein, rosolic acid, and other indicators; but they are neutral to Congo red, methyl violet, phloroglucinol-vanillin, tropæolin, etc. In a liquid containing only hydrochloric acid and such a hydrochloride, therefore, the proportion of "combined hydrochloric acid" can be easily estimated; but the presence of organic acids and acid salts, especially phosphates, renders the process more difficult. Egg-albumin is not precipitated by sodium phosphotungstate, but its hydrochloride is; therefore in neutral solutions albuminous substances are pseudo-bases incapable of precipitation with alkaloid reagents; on addition of acid they become true bases, and can be thrown down. Experiments show that precipitation with neutral alkaloid reagents is a very convenient method of determining the basic equivalent of albuminous bodies, and the method proves that they form as well characterized chemical compounds as other acids and bases. In presence at least of a moderate excess of acid, the amount thereof combined with Witte's peptone can be calculated by the Günzburg reaction or by precipitation with phosphotungstic acid, both processes agreeing very well. And it is accordingly evident that the proportion of combined hydrochloric acid in the contents of the human stomach may be estimated by precipitation with calcium phosphotungstate. The method is very simple and convenient; the reagent is easy to prepare and permanent. F. H. L.

Methods of determining Proteid Nitrogen in Vegetable Matter. G. S. Fraps and J. A. Bizzell. (*Journ. Amer. Chem. Soc.*, xxii., 709.)—The authors have made a large number of experiments to compare the results given by the phosphotungstic acid and bromine methods with those obtained with the Stutzer method. The first of these methods as modified by them consists in stirring 1·4 gramme of the substance with 100 c.c. water, adding the phosphotungstic reagent, and heating the liquid to 60° for fifteen minutes, after which the precipitate is filtered off, washed with water at 60°, and the nitrogen in it determined by the Gunning-Kjeldahl method. The reagent used was a 5 per cent. solution of phosphotungstic acid in 2·5 per cent. hydrochloric acid, 5 c.c. of which were used for every 3 per cent. of nitrogen present. A small excess of reagent is not injurious. If the temperature is lower than 60°, the filtrate is turbid, whilst if it is higher (90° to 100°), part of the nitrogen goes into solution. The results obtained agreed well with those given by the Stutzer method.

The following modification of the bromine method was used: 1·4 grammes of the substance was heated to boiling with 200 c.c. water in a Kjeldahl flask, allowed to cool, acidified with hydrochloric acid, and allowed to stand overnight with a small excess of bromine. The precipitate was then filtered off, washed with bromine-water, returned to the flask, and the nitrogen in it estimated. The results obtained were generally lower than those given by Stutzer's method, and bromine apparently does not precipitate all vegetable proteids.

In three out of four experiments in which the nitrogen was determined by precipitation with zinc sulphate, results were obtained which were slightly higher than those given by the other methods; in the fourth the result was slightly lower. Extraction of the proteids with hot water did not always give concordant results.

The Stutzer method used was as follows: 0·7 gramme of the substance was heated to boiling, or nearly so in the case of starchy materials, for ten minutes with 100 c.c. water, 0·5 gramme of copper hydroxide added, and the liquid stirred; when cold, the precipitate is filtered off, washed with cold water, and nitrogen determined in it. If the substance is rich in phosphates, a little alum solution is added before the copper hydroxide.

The statement that albumoses are not precipitated by this method does not appear to be correct, but in any case the filtrates might be tested with tannic acid.

A. G. L.

Notes on Kühne's Antipeptone. F. Kutscher. (*Berichte*, 1900, xxxiii., 3457-3460.)—The author here supplements the conclusions arrived at in former communications (*Zeit. physiol. Chem.*, xxv., 195; xxvi., 110), and defends his results from the criticisms of Siegfried (*Berichte*, xxxiii., 2851). He again asserts that the residue from albumin, which Kühne regarded as undigested proteid matter, and termed "antipeptone," is a mixture of different amido compounds, etc. On treating fibrin with an equal weight of fresh, finely divided pancreatic tissue, he frequently failed to obtain a biuret reaction after forty-eight hours, and from this concludes that Kühne's "anti" group is not present in fibrin any more than in albumin, and that, therefore, no "antipeptone" can be obtained from them.

C. A. M.

Antipeptone and Amphopeptone. M. Siegfried. (*Berichte*, 1901, xxxiii., 3564-3568.)—In reply to Kutscher's criticisms on his work (*c.f.* preceding abstract), the author lays stress upon the fact that in the experiment on the digestion of fibrin Kutscher continued the digestion for over five weeks, whilst Balke, whose results he criticised, only digested it for four days (*Zeit. physiol. Chem.*, xxii., 255). He therefore considers that Kutscher was not justified in assuming that his product was identical with Balke's antipeptone.

He differs somewhat from the views of Kühne, inasmuch as from the results of his own experiments and those of other observers he considers that antipeptone offers great resistance to the prolonged action of trypsin, but that when the digestion is very active it too is digested.

In conjunction with P. Mühle, the author has attempted to obtain Kühne's amphopeptone by digesting fibrin and Witte's peptone with acid pepsin, and purifying the products by the method recently described by him. He constantly obtained two acids which gave pronounced biuret reaction and had the composition $C_{21}H_{34}N_6O_9$ and $C_{21}H_{36}N_6O_{10}$. He regards these as chemical individuals, and gives the following equation as representative of the relationship of this amphopeptone to the α- and β-antipeptones which he has recently described:

$$C_{21}H_{34}N_6O_9 + H_2O = C_{10}H_{17}N_3O_5 + C_{11}H_{19}N_3O_5.$$

He does not suggest, however, that this reaction occurs in the digestion of amphopeptone by trypsin (see *Berichte*, xxxiii., 2851; *Zeit. phys. Chem.*, xxvii. 335).

C. A. M.

Para-Nitrophenol as an Indicator. L. Spiegel. (*Berichte*, 1900, xxxiii., 2640-2641.)—The author asserts that *p*-nitrophenol can be used as a substitute for methyl orange as an indicator, and that, contrary to Winkler's statement, it is not affected by carbonic acid.

C. A. M.

Chemical and Calorimetric Examination of Fuels. H. Langbein. (*Zeit. f. angew. Chem.*, 1900, 1227 and 1259.)—The author describes an improved calorimeter in which the fuel is burnt with oxygen under a pressure of 20 atmospheres. The chemical composition is ascertained by the analysis of the products of combustion formed in the calorimetric bomb, or in a special bomb of similar construction but smaller. The carbon is determined by absorbing the carbon dioxide in potash bulbs or soda-lime tubes. The water is mostly absorbed by phosphorus pentoxide contained in a small weighed beaker, placed inside the bomb; the remainder is absorbed by causing the products of combustion to pass through calcium chloride tubes before going into the potash bulbs. The sulphur is converted by the combustion into sulphuric acid. The nitrogen present in the oxygen used is converted into nitric acid. To estimate both these the calorimeter is washed out. The solution obtained is boiled to free it from carbon dioxide, and titrated with baryta solution, using phenolphthalein as indicator. Excess of standard sodium carbonate solution is then added; the liquid is allowed to stand some time, then titrated with standard acid, using methyl orange as indicator. The first titration gives the total acid; the second, deducted from the carbonate solution used, gives the nitric acid.

The ash is determined by ignition in a crucible, it then forms a loose powder; but the ash found after a combustion in the bomb is a fused mass, weighing up to 4 per cent. less than that obtained by ignition in a crucible. Moisture is determined by drying at 100° C. under reduced pressure or in a stream of carbon dioxide.

The analytical and calorimetric examinations are made on an air-dry sample. The moisture is determined in a separate portion of the undried fuel, and all the results, analytical and calorimetric, are corrected accordingly. The author gives diagrams of the apparatus, and full details of the method of calculation and the corrections to be made. There are tables giving the results of the examination of some hundreds of different coals and other substances.

A. M.

INORGANIC ANALYSIS.

Volumetric Estimation of Bismuth. G. Frerichs. (*Apoth. Zeit.*, 1900, xv., 859; through *Chem. Zeit. Rep.*, 1900, 383.)—Freshly precipitated bismuth sulphide reacts quantitatively with silver nitrate to form silver sulphide and bismuth nitrate. The sulphide is thoroughly shaken for a short time with a known volume of decinormal silver nitrate, and in a portion of the liquid the excess of reagent is titrated with decinormal ammonium thiocyanate, using iron ammonium alum as indicator.

F. H. L.

The Detection of Copper. A. Bellocq. (*Journ. Pharm. Chim.*, 1900, xii., 363.)—The author's zinc sulphate reagent for the estimation of uric acid (see this vol., p. 75) can be employed as a sensitive test for copper. A litre of water containing 1 drop of a 0·5 to 1 per cent. solution of copper sulphate gives with this reagent, when added until the liquid is strongly alkaline, a slight flocculent precipitate after standing for four to five hours. This is collected in a small crucible, dried, and treated with a slight excess of hydrochloric acid. On now adding ammonium hydroxide the characteristic blue coloration of copper is obtained. C. A. M.

The Analysis of Ferro-Silicon and Silico-Spiegel. Fred. Ibbotson and Harry Brearley. (*Chem. News*, lxxxii., 269.)

Total Carbon.—The powdered alloys are completely burnt by ignition in oxygen, but without the use of any oxidizing reagent like lead chromate.

Graphite.—Two or three grammes of the powdered sample are heated nearly to boiling with 70 to 100 c.c. nitric acid (1·20), and small amounts of hydrofluoric acid gradually added. Glass vessels may be used. The graphite is filtered off on asbestos, and washed in the order named with water, boiling sodium hydroxide, dilute hydrochloric acid, and again with water; it is then burnt in oxygen as usual. Silico-spiegels contain more combined carbon than ferro-silicons. The authors have never found any carborundum.

Silicon.—Hogg's method (*Chem. News*, lxvii., 27) is used as follows: Two grammes of the powdered sample are boiled till decomposed with 50 c.c. hydrochloric acid and 10 to 20 c.c. nitric acid, which will require about fifteen minutes; twice the volume of water is added, and silica filtered off at once, otherwise part of it will dissolve. It is washed with dilute hydrochloric acid, ignited, and weighed. A correction of 0·1 per cent. must be applied for the dissolved silica.

Manganese.—The alloys are readily decomposed with nitro-hydrofluoric acid. In ferro-silicon the amount of manganese is often so low that it may be oxidized with red lead, and titrated. Reddrop and Ramage's method (*Journ. Chem. Soc.*, lxvii., 268) can be applied in all cases: One gramme of the ferro-silicon is dissolved in 30 c.c. nitric acid (1·20), and 1 or 2 c.c. hydrofluoric acid; after cooling, 10 c.c. water and 2 grammes sodium bismuthate are added. The solution is filtered through asbestos, standard hydrogen peroxide solution added, and titrated with $\frac{N}{10}$ permanganate. For silico-spiegels, the solution of the alloy is made up to 100 c.c., 25 c.c. taken and oxidized as above after adding 25 c.c. nitric acid (1·20), and the solution filtered into hydrogen peroxide. Small amounts of hydrofluoric acid do not interfere with the process.

Phosphorus.—The following method is most convenient. To 2 grammes of the alloy 45 c.c. nitric acid (1·20) and 25 to 30 drops of hydrofluoric acid are added, and, after the reaction subsides, another 25 to 30 drops hydrofluoric acid; solution is complete on boiling. Permanganate is then added till a permanent precipitate of manganese dioxide is formed, the solution cleared with ferrous sulphate, graphite filtered off, and, after the addition of 6 or 7 c.c. strong ammonia, phosphorus is precipitated in the filtrate with ammonium molybdate, and weighed as $PbMoO_4$ (*Chem. News*, lxxxii., 55). A. G. L.

Schöffel's Process for estimating Tungsten in Steel. Ernest Bagley and Harry Brearley. (*Chem. News*, lxxxii., 270.)—If Schöffel's process (*Chem. News*, xli., 31) is applied to steels containing chromium, the amount of impurities associated with the tungsten in the residue from the treatment with the double copper salt becomes so large that the method is inaccurate. In such cases the following method, in which hydrochloric acid is added to the copper solution, gives good results, the amount of impurities being very small: 5 grammes of the sample are digested with 50 grammes cuprammonium chloride, 100 c.c. hot water, and 50 c.c. hydrochloric acid, for about half an hour at the boiling-point, with occasional shaking until the copper has dissolved. The residue is filtered off, ignited, silica volatilized with hydrofluoric acid, ferric oxide separated by a sodium carbonate fusion, any chromium in the alkaline filtrate titrated with $FeSO_4$, and the tungsten estimated by difference.

Steels containing only a fraction of 1 per cent. of tungsten should be treated with copper solutions to which only 10 per cent. of hydrochloric acid has been added, otherwise the results are slightly low. If molybdenum is present, part of it may be with the tungsten. A. G. L.

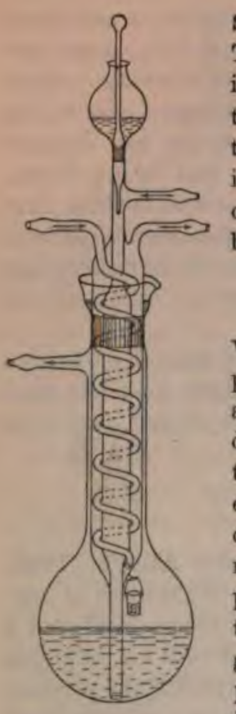
The Estimation of Manganese and Chromium in Tungsten Alloys. Fred. Ibbotson and Harry Brearley. (*Chem. News*, lxxxii., 209.)—Manganese is estimated by dissolving 1 gramme of the sample with 10 c.c. hydrofluoric acid and a few c.c. of nitric acid in a platinum dish, and afterwards adding 2 or 3 c.c. sulphuric acid, transferring to a beaker, oxidizing with lead peroxide, and titrating the permanganate as usual. Results are given which show that hydrofluoric acid does not react with permanganate. This process can only be applied to alloys containing small amounts (about 0·5 per cent.) of manganese. For self-hard alloys, which may contain 10 per cent. of manganese, the chlorate process is better. The dilute hydrofluoric acid hardly attacks glass vessels.

To determine chromium, the sample is digested with sulphuric and hydrofluoric acid, treated with a few grammes permanganate, transferred to a flask, the solution diluted and boiled till all the metal is gone, and the process completed by Stead's method. Another method consists in decomposing the sample with hydrofluoric and nitric acid, adding sulphuric acid, and heating till fumes of the latter come off. The solution is diluted, and either Galbraith's or Stead's process used to estimate chromium. After dissolving the manganese dioxide in hydrochloric acid, tungsten may be estimated by filtering off the WO_3. A. G. L.

Ammoniacal Potassium Sulphate. C. Annato. (*Pharm. Zeit.*, 1900, xlv., 832; through *Chem. Zeit. Rep.*, 1900, 320.)—The author has recently met with two samples of commercial potassium sulphate which passed all the Pharmacopœia tests, but contained ammonia, an impurity that has not hitherto been observed.

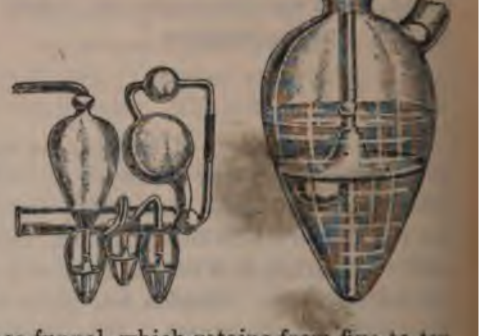
F. H. L.

APPARATUS.



Flask for the Determination of Carbon in Iron and Steel. H. Gockel. (*Zeits. f. agnew. Chem.*, 1900, 1034.)—The apparatus consists of a flask having a side-tube in its neck, into which is ground an arrangement which combines the functions of a condenser, a funnel, and a leading-tube for air. The top of the neck is widened, so that it may be filled with water, in order to render the flask quite airtight. To the bottom of the condenser is attached a small beaker, in which the sample may be placed. A. M.

A Modification of Geissler's Absorption Bulbs. J. Wetzel. (*Ber.*, 1900, xxxiii., [18], 3393, 3394.)—In order to prevent the incomplete absorption of carbon dioxide which sometimes occurs in the elementary analysis of organic substances, notably nitro-compounds, owing to a too rapid evolution of gas, the author employs the modification illustrated in the figure.



Each of the bulbs contains a movable glass funnel, which retains from five to ten small bubbles until they coalesce into a large bubble which can escape into the next bulb. In this way the carbon dioxide is in contact with the potassium hydroxide solution for a considerably longer period. The advantage of this apparatus over the ordinary one is illustrated by an experiment. C. A. M.

REVIEW.

Arsenical Poisoning in Beer-drinkers. By T. N. Kelynack, M.D., and William Kirkby. Baillière, Tindall and Cox.

This is a deeply interesting book, clearly written, and excellently illustrated. Its major portion deals with the clinical appearances observed in peripheral neuritis and the connection of the disease with arsenical beer; its smaller portion with the detection of arsenic and approximate determination of the quantity. It is demonstrated that the consumption of moderate quantities of beer with as little as 0·02 grain of arsenious acid per gallon may be followed by most serious results, and that not only chronic alcoholics, but even children at the breast, may be affected.

The test relied upon by the authors is not one which will commend itself to professional analysts—namely, Gutzeit's—the two best methods, that of Marsh Berzelius and of Reinsch, being dismissed very briefly. It would have been better that part of the work had been written by an experienced analyst.

The book contains an admirable bibliographical appendix. O. H.

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THE ANALYST.

APRIL, 1901.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE monthly meeting of the Society was held on Wednesday evening, March 6, in the Chemical Society's Rooms, Burlington House. The President (Dr. J. Augustus Voelcker, M.A., B.Sc.) occupied the chair.

The minutes of the previous meeting were read and confirmed.

The PRESIDENT read the following reply which he had received to the address from the Council which had been transmitted to His Majesty the King on the occasion of the death of her late Majesty the Queen:

HOME OFFICE, WHITEHALL,
February 15, 1901.

SIR,

I am commanded by the King to convey to you hereby His Majesty's thanks for the loyal and dutiful address of the President and Council of the Society of Public Analysts, expressing their sympathy with His Majesty and the Royal Family on the occasion of the lamented death of her late Majesty Queen Victoria.

I am, Sir,
Your obedient servant,
(*Signed*) CHARLES T. RITCHIE.

The President of the Society of Public Analysts,
Burlington House, W.

The nomination of Dr. T. E. Thorpe, C.B., F.R.S., for election as an Honorary Member of the Society was read for the second time.

Certificates of proposal for election to membership in favour of Messrs. H. Wippell Gadd and A. H. Mitchell, B.Sc., were read for the second time; and a certificate in favour of Mr. John Webster, 21, Park Row, Greenwich, assistant to Mr. J. Kear Colwell, was read for the first time.

Messrs. A. L. H. Garside, R. G. Grimwood, J. C. Umney and H. Rowley were elected members of the Society.

The following papers were read: "The Determination of Dissolved Oxygen in Water in Presence of Nitrites and Organic Matter," by S. Rideal, D.Sc.; "Some Analyses of Oatmeal," by Bernard Dyer, D.Sc.; and "The Detection and Estimation of Preservatives in Milk," by M. Wynter Blyth, B.A., B.Sc.

IS THE BRITISH PHARMACOPŒIA THE LEGAL STANDARD FOR THE PREPARATIONS DESCRIBED THEREIN?

By Alfred H. Allen.

(*Read at the Meeting, February* 5, 1901.)

Probably the great majority of Public Analysts will have observed the rapidly increasing opposition which has existed of late in certain quarters to the claims made that, under the Sale of Food and Drugs Act, the British Pharmacopœia should be considered as a standard or guide for the articles therein mentioned, and the vigorous attempts made in certain quarters to degrade it from the position which it practically held till recently.

Public Analysts owe it to Dr. James Bell that the Pharmacopœia was not made formally an authority under the Sale of Food and Drugs Act. The Select Committee of the House of Commons, upon whose report the Amendment Act of 1879 was based, called two witnesses on the point. One was Mr. Thomas, of the Local Government Board, who expressed strongly the opinion that the British Pharmacopœia should be made the official standard under the Act for the drugs described therein. The second witness was Dr. James Bell, then Principal of the Somerset House Laboratory, who expressed the opinion that the Pharmacopœia was already in practice accepted as the standard for drugs, and that if it were made so more formally it might be troublesome with respect to slight accidental variations. I make this statement from memory, as I have had no recent opportunity of referring to the evidence in question. Dr. Bell having expressed himself satisfied with the then position of the British Pharmacopœia, the question remained in abeyance until a Sheffield pharmacist supplied to the written prescription of a medical man some tincture of opium, which was found on analysis by me to contain only one-third of the opium and a little more than one-half the alcohol present in the British Pharmacopœia tincture. In the face of the evidence given by Mr. G. T. W. Newsholme, at that time the President of the Sheffield Pharmaceutical Society, by Dr. Sinclair White, then Medical Officer of Health for Sheffield, and by myself, the Sheffield stipendiary magistrate decided that, seeing the preparation contained alcohol and opium, it was a tincture of opium, and that deficiency of these ingredients had nothing to do with the question. That decision was not allowed to rest, and the opinion of the High Court of Justice was taken, with the result that the stipendiary's decision was reversed, and the decision in White *v.* Bywater became one of the leading cases on record dealing with the question in point. It was heard before the High Court, Queen's Bench Division, before Chief Justice Coleridge and Mr. Justice A. L. Smith, who decided that the article known as tincture of opium was a well-understood term in the trade as regards the proportion of ingredients; and though the druggist did not profess to sell it according to the British Pharmacopœia, the sample supplied was not that which was asked for.

But this decision was of a limited character; and it has been forcibly contended that the Pharmacopœia is not, and never was, intended to apply to the articles named therein, except when used in dispensing a physician's prescription, or when the

British Pharmacopœia preparation was specifically demanded. This view has been recently endorsed by Dr. John Attfield, the editor of the British Pharmacopœia, who at the Plymouth meeting of the Pharmaceutical Conference, held in 1899, expressed himself as follows: "He hoped that it would be realized by everyone that the Pharmacopœia, notwithstanding the practice of tribunals and the principle on which barristers made speeches, was not a legal standard under the Sale of Food and Drugs Act; and if anyone had the courage to test a case in the superior courts, he was not afraid to forecast that the judges in the superior courts would agree with what he had stated."

But the decision in the case of White *v.* Bywater was largely owing to the evidence brought forward that the British Pharmacopœia tincture of opium was the only one recognised in the trade. If the defence had succeeded in showing that half a dozen qualities of tincture of opium were listed by wholesale druggists, possibly the court might have taken a different view, and held that the purchaser should demand the British Pharmacopœia quality of the tincture.

But I think the existing condition of the law leaves no loophole for an article purchased under the name of one of the preparations of the British Pharmacopœia being otherwise than of British Pharmacopœia quality. Queen Victoria in Council made an order on February 3, 1851, which required that:

"All and singular apothecaries and others, whose duty it is to compound medicines, or distil oils or waters, or make any other extracts within any part of . . . England . . . Wales, or Berwick-on-Tweed, they and every of them, immediately after the Pharmacopœia Collegii Regalis Medicorum Londinensis shall be printed and published, do not compound or make any medicine or medicinal receipt or prescription, or distil any oil or waters, or make any extract, that are or shall be in the said Pharmacopœia . . . mentioned or named in any other manner or form than is or shall be directed, prescribed, or set down by the said book, and according to the weights and measures that are or shall be therein limited, except it shall be by the special direction or prescription of some learned physician in that behalf; and Her Majesty doth hereby declare that the offenders to the contrary shall not only incur Her Majesty's just displeasure, but be proceeded against for such their contempt and offences to the utmost severity of the law."

This Order in Council is still in force.

Further, by the Medical Act of 1862, it is provided that the General Medical Council shall cause to be published a British Pharmacopœia; and Section 3 of the same Act provides that "the British Pharmacopœia when published shall for all purposes be deemed to be substituted throughout Great Britain and Ireland for the several above-mentioned Pharmacopœias, and any Act of Parliament, Order in Council or Custom relating to any of such last-mentioned Pharmacopœias shall be deemed after the publication of the British Pharmacopœia to refer to such Pharmacopœia."

Again, according to the Pharmacy Act of 1868, Section 15, "any person who shall compound any medicines of the British Pharmacopœia, except according to the said Pharmacopœia, shall for every such offence be liable to pay a penalty or sum of £5 . . . but nothing in this Act contained shall prevent any person from being liable to any other penalty, damages, or punishment to which he would have been subject if this Act had not been passed." This last passage appears to tie a pharmacist down to compound his medicines according to the Pharmacopœia's directions *only*, unless by special directions of a medical man.

The foregoing statement represented my view of the question up to November last, when it was originally arranged that I should read a paper on the subject before this Society; but only a few days ago the question has received a stronger and far more authoritative treatment than I could pretend to give it. A week since an appeal case was heard by Mr. Justice Phillimore and Mr. Justice Bruce, sitting in the King's Bench Division of the High Court of Justice. It was a case in which a sample of mercurial ointment was purchased at Skipton, and on analysis by me was found to contain only 12·5 per cent. of mercury, whereas the mercurial ointment of the British Pharmacopœia contains not less than 48·5 per cent. The vendor was convicted, and he appealed to the High Court of Justice on the ground that he could not be convicted under Section 6, as the article was a compounded drug; and further contended that the usual practice was not to supply purchasers with ointment of British Pharmacopœia quality unless ordered by medical prescription. In a very full and lucid judgment Mr. Justice Phillimore held that the British Pharmacopœia was the legal authority for all preparations described therein. Unless those who sympathize with the appellant are prepared to take the case still higher, I presume the recent judgment will stand good for a time at least.

The present situation, to my mind, is very unsatisfactory. Numerous as are the defects in the Pharmacopœia, I have no sympathy with those who try to drag it down from its due position of authority, and degrade it to the standpoint of an irresponsible farrago of second-grade recipes. It is a great misfortune that the Pharmacopœia was not long ago formally recognised under the Adulteration Acts as the standard for drugs described in it; it would then have been easy to have had a schedule appended to the Act specifying certain articles as excluded from the general provision relating to drugs. The course thus suggested was adopted in the Bill laid by this Society before the Select Committee on Food Products Adulteration at their meeting in 1894, and if the advice of the Society had been accepted by the Government, much heart-burning and some injustice would have been prevented. At the present moment the position is most unsatisfactory, and I heartily sympathize with that large and respectable section of pharmacists who have habitually taken the British Pharmacopœia as their guide.

In order to place the law on a more satisfactory basis than exists at the present, I would suggest that a representative committee, including analysts, medical men, pharmacists, manufacturing chemists, and others interested, should endeavour to obtain an interview with the British Medical Council or the Pharmacopœia Committee of that body with the view of the immediate production of an appendix to the British Pharmacopœia, in which appendix titles and undesirable synonyms should be subjected to extensive correction. Subsequent steps would depend on the result of this interview.

DISCUSSION.

Mr. CHATTAWAY thought that, owing to the comparative infrequency of the publication of the Pharmacopœia, its immediate revision would be very difficult to obtain, although such revision was undoubtedly much to be desired. It was to be hoped, however, that the recent High Court decision would have the effect of placing the question upon a more reasonable basis.

Mr. MARTIN PRIEST said that, although the High Court decision just referred to was distinctly a step forward, there still remained the question as to whether, for instance, a grocer selling camphorated oil or beeswax was bound to supply the Pharmacopœia articles. In the case of both camphorated oil and beeswax, it had been held in recent decisions that a grocer was not so bound. Now that the Pharmacopœia was actually established as a legal standard, there would seem to be only two courses open to the compilers of the next edition, namely, either to omit mention of all the characters and tests of the final products, limiting themselves to directions as to quantities and modes of preparation, or to give efficient tests. The former would be a retrograde step, for much assistance was afforded by those of the tests already given, which in justice must be said to be fairly correct. The adoption of the latter alternative would involve considerable research, but would be of very great advantage to all concerned.

Dr. DYER inquired on what authority Mr. Priest contended that the position of a grocer who sold adulterated camphorated oil was in any way different from that of a pharmacist who sold a similar article. The case was quite different from that of beeswax, which was an article used not only as a drug, but for other purposes as well. As a matter of fact, over and over again grocers who had dabbled in selling drugs and had supplied articles not in accordance with the demand of the purchaser, had been properly punished. The case of paregoric afforded a somewhat amusing instance of the liability of grocers dealing in drugs. If a grocer sold paregoric containing no opium, he might be prosecuted under the Sale of Food and Drugs Act; while if he sold paregoric containing opium he might be prosecuted under the Pharmacy Act.

Mr. PRIEST said that a case in reference to camphorated oil was recorded in the *Pharmaceutical Journal* for July 28, 1900, in which such a decision as he had referred to had been given by a magistrate.

Mr. THOMAS TYRER said that it was desirable to distinguish between galenical and non-galenical preparations. There could be no question whatever that a tincture ought to be of the proper alcoholic strength, although the extractive matters might vary within reasonable limits. In the case, however, of an article like Glauber's salt, it would be reasonable enough to stipulate for a given percentage of sodium sulphate —99, 98, or whatever it might be; but it really seemed absurd to covenant that it should have only the "slightest quantity of chlorides, etc." What was the meaning of the term "slightest"? It was highly important that alkaloidal preparations should be what they were represented to be, and he could not imagine that, in the light of modern scientific and technical knowledge, any alkaloid manufacturer would send out articles which were otherwise. Still, it would be wise to stipulate that preparations of quinine, for instance, should contain definite proportions of the alkaloid. But the rest might be omitted: one did not expect to find arsenic in quinine, or selenium or tellurium in Glauber's salt. The essential question seemed to be as to whether there was any evidence of fraud, and for dealing with this a simple statement of the maximum that could be reasonably required under ordinary trade conditions was all that was necessary. As to mercury ointment, he had never made and had never been asked for the weaker kind, and he doubted whether much of it was sent out by the wholesale druggists. At any rate, it was in the vendor's power and his duty to label it properly, and he ought to have done so in his own interest, just as a "poison" label should be affixed to any article requiring it, whether such article was mentioned in the schedule or not. If, as had been suggested in the recent case, mercury ointment of full strength might produce therapeutical effects which were not contemplated, why not let the weaker article be prescribed in the Pharmacopœia? It would only be necessary to employ more of it in any given case. The presence of dangerous quantities of impurities such as arsenic in sodium phosphate was largely due to the prevailing craze for cheapness; but, on the other hand, it was manifestly impossible, in fairness to the purchaser, to insist that articles should be absolutely free from impurities of which, by the utmost refinements of analysis, small traces could be found in anything and everything. He therefore felt that the suggestion concluding the paper was most valuable; but, having regard to the manner in which offers of good work from such bodies as the British Pharmaceutical Conference had been dealt with, he was afraid that its early realization was improbable.

Mr. C. T. TYRER said that it seemed to him obvious that, if the Pharmacopœia was to be a standard, its own standards should be reasonable. Two points seemed likely to be productive of legal complications. The first was that manufacturers, while using the purest materials obtainable, often found themselves quite unable to meet the full requirements of the Pharmacopœia. Secondly, owing to the lack of definiteness which existed in regard to the determination of physical constants, the manufacturer tested in one way, the customer in another, and the analyst to whom the matter might be referred probably in another. He had lately compared certain articles with the standards laid down by seventeen different Pharmacopœias, and had found that, as regards strictness of definition, the British Pharmacopœia was considerably behind in the scale. Nobody would doubt the ability and good faith of its editor, but the system seemed to be radically at fault.

Mr. HEHNER said that if anything were likely to lead to the production of a perfect British Pharmacopœia, it would be the recent legal decision which had been referred to. As long as the Pharmacopœia was not considered seriously, there was no real incentive to make it perfect; but now that, as the result of a High Court decision, it had become definitely the standard by which the articles mentioned in it must be judged, it was to the interest of everybody—and it was bound to come to pass—that its standards should be made to meet the requirements of everybody. Mr. Chattaway had said that there was no chance of its immediate revision; but at the same time those who realized how much work was required to make it perfect would realize also that, if such a result was to be attained even within the next eight years, the work must be taken in hand soon.

Mr. JOHN WHITE said that there had lately grown up a pernicious custom of using the official names or official synonyms of the Pharmacopœia, but with some qualification. Not long ago he had met with a sample bought as camphorated oil, in the case of which the bottle was found to be labelled, in very small letters, with the words, "Camphor Embrocation," and underneath, in large type, "Used as Camphorated Oil." Another example was afforded by the use of the terms, "Sweet Spirit of Nitre, sp. gr. 0·850," and "Sweet Spirit of Nitre, sp. gr. 0·900." Such articles could only be sold with a view to defrauding the public, and it seemed to him that when a new Pharmacopœia was issued this point ought to be properly dealt with. He understood Mr. Tyrer to say that he did not think proceedings should be instituted in respect of any sample of a drug, except in the case of an article which, by reason of some deficiency in strength or quality, was sold with a view to defrauding the public; but surely Mr. Tyrer would admit that in many instances there must be, not only a minimum which ought to be reached, but also a maximum which should not be exceeded. If this principle were not admitted, purchasers of drugs would always be exposed to obvious and improper risks.

Mr. J. B. HARRISON said that in the colony of British Guiana, with which he was intimately connected, the practice was to admit at low rates of duty certain articles when imported as articles of the Pharmacopœia; but if, on analysis by the Government Analyst, such an article was found not to conform within reasonable limits to the requirements of the Pharmacopœia, it was treated as not being in consonance with the Pharmacopœia, and a much higher rate of duty was charged on it. Under those circumstances, at any rate, there could be no advantage in the direction of cheapness from the importation of inferior articles.

Mr. ALLEN said, in reply, in reference to Mr. Chattaway's remark, that his suggestion was that a representative committee should at once obtain an interview with the General Medical Council, or with the Pharmacopœia Committee of that body, urging the desirability of the immediate issue of a revised version of the Pharmacopœia, in which the titles and synonyms should be subjected to extensive correction; or, in the alternative, of an appendix containing the necessary modifications. An appendix to the Pharmacopœia, in fact, was badly needed in any case. This might possibly set the ball rolling, and might be the means of obtaining mitigation of what, he was sure, would be a hardship for a great many classes of persons. It would be a much larger task to carry out the suggestion of Mr. C. T. Tyrer. The revision of the standards and tests of the Pharmacopœia would occupy a considerable period of time, and it was to be hoped that the work involved would be suitably remunerated. He would certainly strongly protest against any unfortunate vendor being brought into a police court simply through a misunderstanding, or through having sold an article which he believed, but had no means of ascertaining, to be correct, as was the case with the unfortunate shopkeeper who sold sweet spirit of nitre without knowing that the strength of the article was likely to diminish on keeping. At the same time, he was rather surprised at the attitude adopted by some speakers and writers on that subject. The vendor should be educated; or, if he could not be educated, the trade in such articles should, as far as possible, be put into the hands of the educated pharmacist, who had means of knowing whether the articles he sold were of the proper quality. To cry "stinking fish" was held to be no excuse for selling unsound meat; and if sweet spirit of nitre was really to be valued by its proportion of nitrous ether, it ought to be kept reasonably up to its proper strength. Pharmacists were too much inclined to be a law unto themselves. One man said that, as far as possible, he took care that all the articles he sold were of British Pharmacopœia quality. Another thought it was sufficient if all the articles with which he made up prescriptions were of British Pharmacopœia quality, the public being supplied with mixtures; and it was this latter class who sometimes got into trouble under the Sale of Food and Drugs Act. He sympathized very greatly with the majority of pharmacists, who, he believed, were doing their very best to maintain the honest traditions of their occupation. He could not help thinking that, if it were really desired, there ought to be a means for a united expression of views to the General Medical Council or its Pharmacopœia Committee. Owing to the recent High Court decision, pharmacists would be in difficulties if it were held that such articles as hard soap were drugs, and must always be in accordance with the Pharmacopœia. Some such articles (such as soda-water, vinegar, etc.) had been removed from the last edition. The time had come when pharmacists would begin to see that the contention that the Pharmacopœia was only intended for use in connection with physicians' prescriptions, and not for everyday use, would not hold water; but he believed that by far the greater number of pharmacists would be only too glad to approach the Medical Council, and to have the assistance of public analysts in attempting to arrive at some reasonable understanding on the question.

The PRESIDENT (Dr. Voelcker) observed that there seemed to be a general feeling of satisfaction that a decision had been given which recognised a certain authority in the matters referred to; but it was clear that the feeling was also general that that authority needed considerable improvement. He agreed with Mr. White in thinking that it was not sufficient to take Mr. Tyrer's view that a departure from the rules laid down should be regarded solely from the point of view of fraud. It certainly seemed very material that people should know the quality or strength of the drugs they used, and insure that the required effect would be adequately produced without being exceeded. It did not seem to be sufficient to say that, if a drug were of half strength only, it was only necessary to use a double quantity of it, for the effect might be very different. It would be much more satisfactory to know, not only that there had been no fraud, but that the production of the proper effect would be insured.

THE ANALYSIS OF SOILS.

By J. ALAN MURRAY, B.Sc.

I SHALL be glad to be allowed to offer a few suggestions in regard to certain proposals contained in a valuable and timely paper—the report of a committee of the Agricultural Education Association—on uniformity in soil analysis, which appeared in the November issue of the ANALYST.

Whatever differences of opinion there may be as to the desirability of uniform processes of analysis, it seems to me that uniformity is essential in the following points:

(*a*) In taking the samples in the field;

(*b*) In manipulation of the coarse samples in the laboratory;

(*c*) In making the solutions;

(*d*) In expressing the results;

and it is upon these points chiefly that I should like to offer some observations on the report of the committee.

With regard to the method of expressing the results, the committee recommend that results should be stated in percentages of the air-dry fine earth. I have, of course, no knowledge of what considerations weighed with the committee in coming to that conclusion; but I notice that the proposal was unfavourably commented on at the meeting, and it is quite clear that results so stated would not be mutually comparable.

It seems to me, however, that there are other and more serious objections to this popular convention. First, it should be kept in view that the results may often have to be interpreted, not by chemists, but by agriculturists; and it is of the greatest importance that they should be stated in some such plain and simple way as to afford at a glance trustworthy information as to the stock of the various substances in the whole soil. It cannot be maintained that a statement of percentage in the fine earth, air-dry or dry, does this: on the contrary, such information can only be deduced by long and troublesome calculations, even when the other necessary data are given, and too often they are not. For example, it may happen that 3, or 30, or 90 per cent. of the total mass of the soil may be made up of stones; the specific gravity of the fine earth itself, and still more the density of packing, varies enormously. It is apparently to be left to the discretion of each chemist whether this information shall be included in his report or not, and without such information a statement of percentages means little or nothing.

Secondly, the figures showing the percentages of the most important ingredients —phosphoric acid, potash, etc.—in the fine earth are, as a rule, inconveniently small, and the importance of those of the third decimal place may easily be unappreciated. Thus, the percentage of total phosphoric acid is often represented by figures of smaller value than 0·1, and that of the "available" portion by figures less than 0·01; and yet 0·001 per cent., which to many people might seem almost a negligible quantity, would, in most cases, be equivalent to about 20 pounds of phosphoric acid per acre, and would require over 1½ cwt. of superphosphate to supply it as manure.

Thirdly, the total amount of water present in a soil at the time of sampling is naturally extremely variable; and though it depends largely upon circumstances beyond control, and which may for many purposes be regarded as accidental, it is by no means devoid of interest and importance, especially if the rainfall in the district for the few days immediately preceding sampling be known or can be ascertained. The fact that it cannot conveniently be included with the other substances in the total of 100 per cent. must therefore be regarded as an objection to the plan of rendering results in terms of percentages rather than as a justification for ignoring the water altogether.

Finally, a plant's chances of obtaining its necessary supplies of any substance depend, not upon the amount of that substance in a unit of mass, but in a unit of volume; and, consequently, since a given volume of one soil may in extreme cases weigh perhaps twice as much as an equal volume of another, a simple statement of percentages does not afford a reliable basis for comparison of degrees of fertility of different soils.

The remedy for this last-mentioned objection appears simple—namely, to express the results of analyses in terms of the quantities per unit of volume of the soil *in situ*.

Thus, the quantities of potash, of phosphoric acid, etc., in a given volume of soil form separate statements independent of each other, of the quantities of water and of everything else, so that the quantity of water may be included or omitted without in any way affecting the figures relating to the other ingredients.

A report in these terms would yield, on multiplication of the figures by some simple factor, an expression of the quantities per acre, and by choice of suitable units it might be rendered in figures of any size we please.

The question of the particular units to be chosen is, of course, a matter of minor importance; but, all things considered, it would probably be hard to find any with more to recommend them than ounces per cubic foot. These are common English standards, with which every farmer is familiar; they are easily converted to pounds and acres respectively, and they produce figures of about ten times the magnitude of those expressing the same results in terms of percentages, so that it would only be necessary to carry the figures to the third decimal place instead of the fourth. Even the fact that the measurements would in most cases be made in the units of the metric system offers no obstacle, for, as it happens, the relation of the gramme to the ounce is practically the same as that of the cubic decimetre to the cubic foot; and hence, if results expressed in terms of grammes per cubic decimetre be simply read as ounces per cubic foot, the error involved is not greater than 1 in 700—a quantity which for practical purposes might be neglected. In other words, the exact relation between grammes per cubic decimetre and ounces per cubic foot is as 700 to 701, which gives the factor 1·00143 for converting the former to the latter in cases where great accuracy is required.

In order to give effect to the suggestion, however, it would be necessary to determine the mass of some definite volume of the soil *in situ* by means of a specially constructed sampler, as recommended by the committee, or in some other way. The sampler, if used, need not be exactly a cubic foot or a cubic decimetre, but its volume content must be accurately known. A cylinder 9 inches deep by 3 inches in diameter holds approximately a cubic decimetre, and is a convenient instrument in many ways, though it cannot be used in every case. A wider one is necessary for very stony soils, and a longer or shorter one where the soil is to be sampled at different depths.

The depth of soil sampled is very important in this connection, as it determines the factor for converting ounces per cubic foot into pounds per acre. Thus, if the depth be 12 inches, the factor will be $43560 \times \frac{1}{16} = 2722$ nearly; but if the depth be 9 inches only, the surface area in the cubic foot will be greater, and the factor in that case would be $43560 \times \frac{1}{16} \times \frac{3}{4} = 2042$ very nearly. In the course of the discussion which followed the reading of the paper, Dr. Dyer in speaking of the proposal of the committee that soils should be sampled to a depth of 9 inches, mentioned that Sir Henry Gilbert now considered about 8 inches would, as a rule, be more suitable, and he suggested one-fifth of a metre as a convenient international standard. I do not propose to discuss in this connection what depth would be most suitable under ordinary or any circumstances; but it occurred to me at the time that, if the fifth of a metre were adopted, it would fall in very conveniently with the suggestion I have ventured to put forward.

It will, further, be seen that the proposal to state the results in quantities per unit of volume of the soil *in situ* would involve a large departure in the methods of treating the samples in the laboratory from those at present commonly adopted and recommended by the committee. Thus, a sample taken in the manner and for the purpose referred to might be accepted as representative of the soil of the field or part of the field, or it might be rejected as the reverse; but in either case it would have to be accepted or rejected as a whole, and its properties would have to be determined in the mass as it occurs—*i.e.*, without any preliminary process of picking or sifting, or any other arbitrary method of separating the fine and coarse particles. For it is evident that a statement that a cubic foot of soil *in situ* contains a certain quantity of any ingredient soluble in concentrated or dilute acids implies that a representative portion of such soil as it occurs *in situ* has been submitted to the action of the solvent.

It may be argued, of course, that the coarser particles of the soil do not, within a measurable time, affect its chemical properties; but this, even if it were true, is not a satisfactory reason for excluding these particles from the action of the reagents, but quite the reverse. The roots of plants are commonly found adhering very closely to the stones. It is possible that the plants derive merely mechanical advantage in this way, though the facts, so far as they are known, point in the opposite direction; but if the roots extract nothing from the stones, then neither will those reagents with which we endeavour to imitate their action, if they do imitate it; and if the roots do obtain nourishment from the stones, then the stones should be treated exactly like the other particles of the soil.

The arbitrary use of a sieve of certain dimensions for the purpose of discriminating what is fine and what is coarse material—*i.e.*, what is soil and what is not—probably does not commend itself greatly even to the members of the committee who recommend it, and it is certainly much easier to excuse it as necessary than to defend it as reasonable and satisfactory. Probably few persons could be found to approve of manipulating a sample of any other kind in a similar way; but without doubt, in the case of soils, the line must be drawn somewhere. Thus, a sample might be drawn containing a stone as big as a man's head, and perhaps only as much fine stuff as would fill a teacup; a sample might be drawn containing the stump of a tree; but such samples would not be representative, and would therefore be rejected altogether. It may be taken for granted, then, that some method of deciding what substances might be legitimately included as representative in a sample of soil is indispensable, but it does not follow that it should be the arbitrary one at present in general use. On the contrary, it seems to me that each case should be judged separately and on its own merits, on the principle that all those stones, etc., of whatever size, which are more or less uniformly distributed throughout the mass of the soil should be regarded as essential parts of it, and that those which are exceptional in point of size or in other respects should be regarded as accidental, when, of course, no sample containing them could be accepted.

It is to be observed that this principle, should it be accepted, does not leave the decision to the somewhat haphazard method of simple inspection—though even that would in most cases form a sufficiently reliable guide—because in estimating the mass of a cubic foot it is always necessary to make several trials, and, under ordinary circumstances, the weights will usually be found to differ only by a few grammes; but should an exceptionally large stone or other accidental substance be included in one of the samples so drawn, the weight of that one will be found to differ very widely from the average of the others, and the sample would therefore be rejected.

The plan I have adopted in taking a portion of the soil for analysis has been to disintegrate the various masses of wet soil taken out by the sampler with the fingers, mix them as thoroughly as possible, and bring the lot together in a heap; then with a long-bladed spatula divide off such a portion as I reckoned would constitute a fair sample of the heap, and sweep it into a weighed beaker, and weigh again to find the quantity of soil taken. In most cases I found that about 200 grammes was sufficient, but under certain circumstances it was necessary to take larger quantities. It is, of course, necessary at the same time to set aside a portion to air dry, and afterwards to dry it thoroughly, in order to determine the moisture. Any errors which might be involved by the rougher method of sampling would probably be more than compensated by the difference in the size of the portions taken. The exact nature of the difficulties to be encountered and the best methods of dealing with them could, however, probably be most satisfactorily dealt with by discussion in committee.

I have found it easier to get the solvent thoroughly mixed with the large mass of soil, and in one case I found a considerably larger proportion of "available" phosphoric acid extracted when the sample was treated in the undried condition. Dehydration of hydrated phosphates might possibly account for this; but whatever was the cause, the point is one of great importance, and I regret that I have been so far unable to pursue my investigations in regard to it.

I think the scheme of the committee would also be greatly improved if it included a recommendation that the amount of humus should be deduced from the determinations of the organic carbon. The loss on ignition, after drying at 100° C., can rarely correspond to the amount of humus, for it includes the combined water of clay, etc. In a soil which I examined recently I found only about 0·4 per cent. of organic carbon and no carbonates at all, and yet this sample lost nearly 5 per cent. on ignition after drying. In the course of certain other experiments, I found that, as a rule, the organic carbon corresponds to about half the weight of the humus present; but the relation is probably not constant, and the matter requires further investigation.

REGULATIONS FOR MILK AND CREAM.

REPORT OF THE DEPARTMENTAL COMMITTEE APPOINTED BY THE BOARD OF AGRICULTURE TO INQUIRE INTO AND REPORT UPON THE DESIRABILITY OF REGULATIONS (UNDER SECTION 4 OF THE SALE OF FOOD AND DRUGS ACT, 1899) FOR MILK AND CREAM.

AT the beginning of 1900 the Board of Agriculture, in pursuance of their powers under Section 4 of the Sale of Food and Drugs Act, 1899, appointed a Departmental Committee to inquire and report what regulations, if any, should be made in the case of milk and cream for determining what deficiency in the normal constituents, or what addition of extraneous matter, should be held to raise a presumption that the milk or cream was not genuine.

This Committee consisted of the following members: Lord Wenlock, G.C.S.I., G.C.I.E. (Chairman); Mr. George Barham (Dairy Supply Co., London); Mr. George Cowan (Dairy Farmer, Wigtownshire, N.B.); Major Craigie (Assistant-Secretary Board of Agriculture); Mr. S. W. Farmer (Dairy Farmer, Little Bedwyn, Wilts); Dr. Shirley F. Murphy (Medical Officer, London County Council); Professor Thorpe, F.R.S. (Principal Chemist, Government Laboratories); Dr. J. A. Voelcker (Consulting Chemist, Royal Agricultural Society of England); with Mr. R. H. Rew (Board of Agriculture) as Secretary.

The Report of the Committee was made to the Board of Agriculture, and, having been presented to both Houses of Parliament, has now been published, together with the Minutes of Evidence taken before the Committee.

The Committee held fifteen sittings for the purpose of taking evidence, and examined forty-nine witnesses. Of these, fifteen were analysts, and included names so well known as Mr. A. H. Allen, Mr. E. J. Bevan, Sir Charles Cameron, Mr. C. E. Cassal, Dr. Dyer, Mr. W. W. Fisher, Dr. Alfred Hill, Mr. G. Lewin, Mr. F. J. Lloyd, and Mr. H. D. Richmond. Four witnesses were Medical Officers of Health (for Birmingham, Glasgow, Manchester and Reading respectively); while the views of the Dairy Trade, the milk-producing farmers, agricultural societies, and Dairy Farmers' Associations, were all fully represented, and the evidence comprised in a Blue Book of 450 pages.

The Report itself is in two parts, the majority report, signed by seven of the eight members, comprising thirty-three pages. It is subject, however, to a reservation by one of the signatories, Mr. S. W. Farmer, who is in favour of "seasonal limits." The minority report, which extends to thirty-six pages, and traverses the whole ground of the majority report, is signed by Mr. George Barham alone.

The subjects, considered in order, were: (1) Whole milk; (2) skimmed or separated milk; (3) condensed milk; (4) cream. There was a general consensus of opinion that there ought to be regulations laid down for determining when milk and cream should be considered genuine, though some few witnesses, mostly representatives of dealers in and distributors of milk, thought that no such regulations were necessary. The variability of the so-called "standards" adopted in different towns and by different analysts was brought out, and a general desire expressed that uniformity should be arrived at.

The Report next deals with the various difficulties standing in the way of fixing limits. "Minimal limits," it may be said, is the sense in which the Committee interpret the expression "standard" now so generally used. The causes of variability in the quality of milk, viz., the land, the method of feeding (the influence of which is not regarded as definite), the breed, condition, period of lactation, interval between times of milking, the time of year, etc., are successively dealt with. The "standards" adopted in other countries are set out, but are not considered to have much bearing on the subject immediately in hand. The question is then discussed very fully whether "local limits," applying to particular districts, or "seasonal limits," applying to particular times of the year, could be adopted; and whether milk could be "graded" according to quality, and sold as of certain grades and at corresponding prices. The Report shows the impracticability, in the Committee's opinion, of all these suggestions, though the question of "seasonal limits" called for, as it merited, very careful consideration, and it was on this point alone that Mr. Farmer, one of the Committee, found it necessary to append his reservation when signing the general Report. The general feeling of the Committee was that though; as a rule, it would be in the months of May and June that milk would be somewhat lower in quality, yet there might be great variation in particular years, and that any concession given for certain months and particular cases would lead to much greater difficulties as regards the administration of justice, while the difficulties of the producer could be met partly by his own efforts in insuring regularity and uniformity of supply, and partly by the reasonable application of the proposed "limits" by those responsible for administering the Act.

In dealing next with the particular "limits" to be adopted, the Committee have clearly been guided mainly by the views of those who—such as the analysts—have no personal interest in the matter, but are conversant with the chemical examination of milk as supplied to the public and during all times of the year. It is pointed out that while farmers generally favoured a limit of 3 per cent. of fat, and in several cases expressed a preference for a higher limit, the representatives of the traders in milk, almost without exception, advocated a low "standard" or none at all. A large number of statistical tables were put in giving the results of analyses of samples of milk taken over considerable periods and as supplied to different dairies, creameries, etc., over the country, and the Committee arrived at the conclusion that the milk produced for sale, as such, in this country contains, on an average, from 12·5 to 12·8 per cent. of total milk-solids, comprising from 3·7 to 4 per cent. of milk-fat. After weighing carefully the evidence given them, and especially that of the analysts, they came to the conclusion that any milk the total milk-solids of which fall below 12 per cent. should be made the subject of "further inquiry." If it should then be found to give less than 3·25 per cent. of milk-fat, it is recommended that a presumption be raised—until the contrary is proved—that it has been mixed with separated milk or water; and, if the non-fatty milk-solids are less than 8·5 per cent., the presumption that it has been mixed with water shall arise.

Accordingly, the governing factor in the first instance is to be that of a limit of

12 per cent. of total milk-solids, and it is only when a milk fails to give this percentage that it is further inquired into, and the other factors, viz., 3·25 per cent. of fat and 8·5 per cent. of non-fatty solids, come into force.

It is well that this should be clearly explained, as a good deal of misapprehension appears to exist on the point.

The Report next puts out certain suggestions for the better administration by local authorities of their powers under the Act, whereby the interests of the honest trader may be safeguarded. Uniformity in methods of taking samples and of analysis is advocated, as well as the official standardizing of "mechanical test" bottles.

In regard to hand-skimmed and machine-separated milk the Committee think that there is no necessity to make a distinction between them, but that both should be sold under a "limit" of 9 per cent. of total milk-solids. They further give their opinion that it would be very desirable to identify or "ear-mark" all separated milk. The subject of Condensed Milk also occupied the attention of the Committee, and there was strong expression as to the necessity of limiting the amount of sugar used in these preparations, and to the securing that the milk, when diluted with water to the extent stated on the label, should be judged by the limits for ordinary milk. It was recommended, therefore, that condensed milk should contain not less than 10 per cent. of fat or 25 per cent. of solids-not-fat.

Lastly, as regards cream, the Committee did not advocate the fixing of any definite limits of fat, etc., but they considered that cream might well be sold as of different qualities, at corresponding prices. They advised, however, that any artificial thickening of cream should be considered as an adulteration.

To the five main recommendations which they give in their Report the Committee append seven minor suggestions as expressions of opinion, and the whole are set out as follows:

RECOMMENDATIONS.

114. The Committee beg to make the following recommendations:

I. That regulations under Section 4, of the Food and Drugs Act, 1899, be made by the Board of Agriculture with respect to Milk (including condensed milk) and Cream.

II. (*a*) That in the case of any milk (other than skimmed, separated, or condensed milk) the total milk-solids in which on being dried at 100 degrees Centigrade do not amount to 12 per cent. a presumption shall be raised, until the contrary is proved, that the milk is deficient in the normal constituents of genuine milk.

(*b*) That any milk (other than skimmed, separated, or condensed milk) the total milk-solids in which are less than 12 per cent., and in which the amount of milk-fat is less than 3·25 per cent., shall be deemed to be so deficient in milk-fat as to raise a presumption, until the contrary is proved, that it has been mixed with separated milk or water, or that some portion of its normal content of milk-fat has been removed. In calculating the percentage amount of deficiency of fat the analyst shall have regard to the above-named limit of 3·25 per cent. of milk-fat.

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(c) That any milk (other than skimmed, separated, or condensed milk) the total milk-solids in which are less than 12 per cent., and in which the amount of non-fatty milk-solids is less than 8·5 per cent., shall be deemed to be so deficient in normal constituents as to raise a presumption, until the contrary is proved, that it has been mixed with water. In calculating the percentage amount of admixed water the analyst shall have regard to the above-named limit of 8·5 per cent. of non-fatty milk-solids, and shall further take into account the extent to which the milk-fat may exceed 3·25 per cent.

III. That the artificial thickening of cream by any addition of gelatin or other substance shall raise a presumption that the cream is not genuine.

IV. That any skimmed or separated milk in which the total milk-solids are less than 9 per cent. shall be deemed to be so deficient in normal constituents as to raise the presumption, until the contrary is proved, that it has been mixed with water.

V. That any condensed milk (other than that labelled "machine-skimmed milk" or "skimmed milk," in conformity with Section 11 of the Food and Drugs Act, 1899) in which either the amount of milk-fat is less than 10 per cent., or the amount of non-fatty milk-solids is less than 25 per cent., shall be deemed to be so deficient in some of the normal constituents of milk as to raise the presumption, until the contrary is proved, that it is not genuine.

115. The Committee beg further to submit the following expressions of opinion on points raised before them in evidence:

(a) That it is desirable to call the attention of those engaged in the administration of the Food and Drugs Acts to the necessity of adopting effective measures to prevent any addition of water, separated or condensed milk, or other extraneous matter, for the purpose of reducing the quality of genuine milk to any limits fixed by regulation of the Board of Agriculture.

(b) That it is desirable that steps should be taken with the view of identifying or "ear-marking" separated milk by the addition of some suitable and innocuous substance, and by the adoption of procedure similar to that provided by Section 7 of the Food and Drugs Act, 1899, in regard to margarine.

(c) That it is desirable that, so far as may be found practicable, the procedure adopted in collecting, forwarding, and retaining pending examination, samples of milk (including condensed milk) and cream under the Food and Drugs Acts should be uniform.

(d) That it is desirable that, so far as may be found practicable, the methods of analysis used in the examination of samples of milk (including condensed milk) or cream taken under the Food and Drugs Acts should be uniform.

(e) That it is desirable in the case of condensed milk (other than that labelled "machine skimmed milk" or "skimmed milk," in conformity with Section 11 of the Food and Drugs Act, 1899), that the label should state the amount of dilution required to make the proportion of milk-fat equal to that found in uncondensed milk containing not less than 3·25 per cent. of milk-fat.

(*f*) That it is desirable in the case of condensed whole milk to limit, and in the case of condensed machine-skimmed milk, to exclude, the addition of sugar.

(*g*) That the official standardizing of the measuring vessels commercially used in the testing of milk is desirable.

Mr. S. W. Farmer, though he signs the Report generally, makes a reservation, as stated, in regard to "seasonal limits." He holds that 12 per cent. of total solids is too low for six months of the year, and that 3·25 per cent. of fat is too high for the spring months. He would like to see seasonal limits of 3 per cent. of fat and 8·5 per cent. of non-fatty solids for March, April, May and June, and 3·25 per cent. of fat and 8·5 per cent. of non-fatty solids for the rest of the year.

Mr. George Barham, in his minority report, differs entirely from his colleagues on the main points regarding whole milk. He would advocate a limit of total milk-solids of 11·75 per cent., with 3 per cent. of fat in the months of July to February inclusive, lowered to 2·75 per cent. of fat in the months of March to June inclusive, with a limit of 8·5 per cent. of non-fatty solids, in both cases alike.

In the case of skimmed or separated milk, Mr. Barham recommends a limit of 8·75 per cent. total solids instead of the 9 per cent. recommended in the majority report.

The Report, price 7½d., can be obtained from Messrs. Eyre and Spottiswoode, East Harding Street, Fleet Street, E.C.; or other Government publishers; as also the Minutes of Evidence, Appendices, etc., price 3s. 8d.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

Ramschen's Method of estimating Fat in Milk. **R. Lézé.** (*Repert. Pharm.*, 1901, xiii. [3], 1; through *Chem. Zeit. Rep.*, 1901, 52.)—This process has been investigated by Fouard, and the present author considers it very useful. The reagent consists of 8 grammes of potassium hydroxide, 10 c.c. of ordinary ammonia, 55 c.c. of ethyl alcohol, and 15 c.c. of amyl alcohol. When the potash has dissolved, the whole is diluted with ammonia to 100 c.c. Thirty-six c.c. of milk and 10 c.c. of the reagent are brought into a flask holding 50 or 60 c.c., the neck of which is graduated. The flask is placed on a boiling water-bath, and gently agitated at intervals for about twelve minutes. Hot water is then added till the liberated fat collects in the graduated portion of the neck, and its volume is read off at a temperature of 40° C. At this point the specific gravity of butter-fat is about 0·90. F. H. L.

The Composition of the Juice of Certain Fruits. Truchon and Martin-Claude. (*Journ. Pharm. Chim.*, 1901, xiii., 171-176.)—The authors have analysed the juices of several kinds of fruit intended for the manufacture of syrups, etc., and have obtained the following results, expressed in grammes per litre :

Juice.	Specific Gravity	Invert Sugar.	Saccharose.	Saccharimeter, degrees.		Acidity as Tartaric Acid.	Substances precipitated by Alcohol.	Mineral Matter.	K_2O.	P_2O_5.	Citric Acid.	Tartari Acid.
				Before Inversion.	After Inversion.							
Cherry (early)	1040·4	83·60	—	− 9°	− 9°	4·95	3·40	3·00	0·44	0·33	slight trace	presen
Cherry (in season) ...	1055·4	96·49	—	− 16° 5′	− 16° 5′	8·46	2·40	3·88	0·97	0·21	—	—
Early Strawberry ...	1026·2	45·18	—	− 7°	− 7°	9·15	10·00	5·96	0·46	0·60	present	trace
Strawberry (in season) ...	1048·2	99·98	—	− 21°	− 21°	11·52	13·80	5·72	0·97	0·26	—	presen
Raspberry ...	1050·3	88·19	—	− 15°	− 15°	17·82	9·60	4·32	0·86	0·32	—	—
Gooseberry : Red... ...	1040·0	63·68	—	− 11°	− 11°	28·50	8·60	5·40	1·25	0·47	—	—
Gooseberry : Green ...	1049·8	87·43	—	− 15° 3′	− 15° 3′	25·65	7·20	4·44	1·01	0·25	—	—
Black Currant	1065·5	116·60	—	− 25°	− 25°	31·44	10·80	7·20	1·63	0·66	—	presen
Peach.. ...	1054·0	33·50	19·80	+ 2° 5′	− 14°	6·84	7·60	4·70	0·76	0·46	—	—
Pear	1055·0	85·80	—	− 10° 2′	− 10° 2′	2·04	2·60	3·56	1·68	0·16	—	—
Quince ...	1048·0	75·90	—	− 7° 4′	− 7° 4′	9·60	4·60	4·20	1·81	0·37	—	presen
Apple... ...	1068·0	102·80	6·60	− 8°	− 13° 5′	7·44	6·80	3·72	2·09	0·19	—	—

Analyses of the entire fruits (pulp and juice) gave the following results, calculated on 100 grammes :

Fruit.	Invert Sugar, per Cent.	Saccharose, per Cent.	Saccharimeter, degrees.		Mineral Matter, per Cent.	K_2O, per Cent.	P_2O_5, per Cent.	Citric Acid.	Tartaric Acid.
			Before Inversion.	After Inversion.					
Apricots ...	2·64	4·15	+ 29° 6′	− 4° 8′	0·59	0·126	0·05	present	present
Greengages ...	8·80	0·80	− 7	− 14	0·57	0·115	0·06	—	trace
Mirabelles ...	6·57	3·04	+ 5	− 20	0·59	0·217	0·07	—	—

None of the juices examined contained glucose.

In testing for salicylic acid, 100 c.c. of the sample were diluted with the same volume of water, and about 2 c.c. of a concentrated solution of ferric chloride added. The liquid was shaken until homogeneous, about 10 grammes of calcium carbonate added, the precipitate allowed to settle, the filtrate extracted with ether, and the residue from the extract tested with dilute ferric chloride solution. Without this treatment, several of the juices, notably that of the strawberry, gave colour reaction in the absence of salicylic acid.

Of the natural colouring matters, only that of the peach was soluble in ammoniacal amyl alcohol, yielding a yellowish-red colour, which, however, did not dye silk.

The authors have proved that the natural colours cannot be confounded with coal-tar or other added colouring matters, such as archil, logwood, cochineal, etc.

C. A. M.

The Composition of Belgian Butter. J. Wauters. (*Bull. de la Soc. Belge*, 1900, xiv., 453-470.)—In discussing the new Belgian law for the prevention of the adulteration of butter with margarine, the author gives a résumé of the investigation made by the Belgian Government as to the composition of the butter manufactured in that country.

The butter manufactured, in most cases under the supervision of an inspector, by fourteen co-operative dairies and thirty-three wholesale firms, representative of every part of Belgium, was analysed every fifteen days for a year (1897-98) by two different chemists. The results thus obtained were as follows:

In the case of the fourteen co-operative dairies, 330 samples were analysed between October, 1897, and October, 1898, and the following variations observed: Specific gravity, 0·864 to 0·8686; refractive index (Abbe-Zeiss), 40·2 to 46·7; Meissl value, 26·07 to 34·50; Hehner value, 85 to 89·65.

During the same period 755 samples taken from the wholesale firms were analysed, with the following results:

Specific gravity	0·8636 to 0·8684	
Abbe-Zeiss index	40 to 47	
Meissl value ...	26 to 36·85 (one 40·35)	for 693 samples.
	25·0 to 25·84	for 26 ,,
	24·0 to 24·96	for 16 ,,
	23·08 to 23·84	for 9 ,,
	19·8 to 23·0	for 11 ,,
Hehner value ...	85·25 to 89·0	for 609 ,,
	89·0 to 89·90	for 139 ,,
	90·00 to 91·0	for 7 ,,

Similar investigations were also made with the butter from special districts, and with that of special firms which had given abnormal results, the analyses being continued to the beginning of 1900. The majority of these samples gave results analogous to the majority of those shown in the preceding tables.

The Commission came to the conclusions that as a rule the butters giving abnormal figures were derived from small dairies; that these abnormalities were of most frequent occurrence from August to December; and that they disappeared during the months of March, April, and May. C. A. M.

The Detection of Cinnamic Acid in the Presence of Benzoic Acid. A. Jorissen. (*Ann. de Chim. Anal.*, 1901, vi., 41-43.)—When cinnamic acid is oxidized by a suitable reagent, it is converted into benzaldehyde, which can readily be recognised by its odour.

In the case of official benzoic acid, 0·1 gramme is dissolved in 5 c.c. of boiling water, and 0·1 gramme of potassium permanganate added.

A second test of the same kind is based on the combined action of uranium salts and light upon cinnamic acid. A few decigrammes of the sample are mixed with a few c.c. of a 5 per cent. solution of uranium acetate or nitrate, which are then exposed to the light in a stoppered flask. After some minutes a brown deposit of uranous oxide (?) is formed, and the odour of benzaldehyde will be observed.

C. A. M.

Valuation of Jalap Tubers. O. Schweissinger. (*Pharm. C. H.*, 1901, xlii., 1; through *Chem. Zeit. Rep.*, 1901, 14.)—Ten grammes of the finely-powdered roots are extracted with spirits of wine for twenty-four hours at a temperature of 30° C., shaking at intervals. Fifty c.c. of the liquid are drawn off, and the solvent is evaporated. The resin is then washed with warm water as long as colouring-matter is removed, and the residue is dried. The results are concordant. F. H. L.

TOXICOLOGICAL ANALYSIS.

A Test for Picrotoxin. S. S. Minovici. (*Ann. Pharm.*, 1901, vii., 1; through *Chem. Zeit. Rep.*, 1901, 52.)—Anisaldehyde is a very delicate and characteristic reagent for picrotoxin, indicating its presence not only when alone, but also in the starchy mass of the fruit. A drop of sulphuric acid is added to the sample, and after two minutes, when the saffron-yellow colour has become distinct, one drop of a 20 per cent. solution of anisaldehyde in absolute alcohol is introduced. A dark-blue violet ring is produced, which soon changes to a permanent blue. The test can also be applied to dilute solutions in chloroform, alcohol, or water; but the liquids should be warmed. The limit of sensitiveness is 1 : 2,000, or even 1 : 5,000. Many other glucosides and alkaloids give obscure and unimportant colour reactions with the reagent; while convolvulin yields a cherry-red, saponin a red-brown, aconitine a pale rose, and veratrine a blue-red or indigo-blue tint. F. H. L.

Identification of Tropine. S. Vrevin. (*Ann. Pharm.*, 1900, vi., 511; through *Chem. Zeit. Rep.*, 1901, 12.)—The most characteristic property of tropine is the ease with which it yields crystalline double salts, especially double chlorides with platinum and gold. With cadmium potassium iodide in a weak acid solution tropine gives a crystalline precipitate consisting of hexagonal plates. The compound is relatively soluble in water, and strong solutions are necessary to obtain it; it melts above 200° C. to a clear liquid. With a faintly acid solution of phosphomolybdic acid tropine yields a yellowish precipitate of microscopic needles entangled together. On warming, this compound turns green and then decomposes without melting. These two reactions readily distinguish tropine from the four principal mydriatic alkaloids of the *Solanaceæ*, as they give with cadmium potassium iodide either amorphous precipitates or crystalline ones of quite different appearance, and with phosphomolybdic acid they yield amorphous precipitates only. F. H. L.

ORGANIC ANALYSIS.

On the Separation of Oleic Acid from other Unsaturated Acids. J. Lewkowitsch. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1901, iv., 62.)—The author, replying to Farnsteiner (*Zeit. für Untersuch der Nahr. und Genussmittel*, 1900, iii., 537-539; ANALYST, 1900, 293), states that in 1899 experiments were undertaken in his laboratory with the object of testing Farnsteiner's method, and that these proving unsatisfactory, he privately communicated the results to Farnsteiner. At the same time he sent him a sample of the oleic acid he produces on a large scale for examination by his method. As Farnsteiner found in this sample only 23·5 per cent. of oleic acid, the author considers his strictures upon Farnsteiner's method were fully justified.

H. H. B. S.

A Reply to the foregoing Remarks of Lewkowitsch. K. Farnsteiner. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1901, iv., 63-65.)—The author contends that Lewkowitsch has misunderstood his meaning. His method was only intended as a test for the presence of oleic acid, and was so described in the title of the paper. He never at any time claimed for it that it was a complete method adapted for general use for the separation of oleic acid from other unsaturated acids. He, however, explains the fact that he found only 23·5 per cent. of oleic acid in the sample sent him by Lewkowitsch by supposing that the process used by him in its preparation—heating mutton-fat under pressure with lime and water—does not yield a pure product.

H. H. B. S.

The Characters of Oil of Akee. W. Garsed. (*Pharm. Journ.*, 1900, 691.)—The oil is a yellow non-drying fat consisting, at ordinary temperatures, of a liquid and of a solid granular portion. It has a peculiar odour, and a somewhat unpleasant taste. It consists of about 50 per cent. liquid, and 40 per cent. solid glycerides, and 10 per cent. of free fat-acids. The acids contain oleic acid, and either a mixture of, or near homologues of, palmitic and stearic acids. In the following table the constants of akee oil are compared with those of olive oil and palm oil:

	Akee Oil.	Palm Oil.	Olive Oil.
Specific gravity	99°-100° C., 0·857	98°-99° C., 0·8586	15·5° C., 0·914-0·917
		(Water at 15·5° C. = 1.)	
Melting-point	25°-35°	27°-42·5°	2·5°
Solidifying-point	20°	21°-27°	+2° to −4°
Hehner value	93	94·2-97	95·4
Saponification value	194·6	196·3-202·5	185-196
Reichert value	0·9	0·5	0·3
Iodine value	49·1	51-52·4	81·6-84·5
Acid value	20·1	—	—

Mixed Fatty Acids.

	Akee Oil.	Palm Oil.	Olive Oil.
Specific gravity at 99° C.	0·8365	0·8369	0·843
		(Water at 15·5° C. = 1.)	
Melting-point	42°-46°	47·7°-52°	22°-26°
Solidifying-point	40°-38°	44°-45°	21°-24°
Saponification value	207·7	206·5-207·3	
Iodine value	58·4	—	86·1-90·2

A. G. L.

Test for Sesamé Oil in Vegetable and Animal Oils. Tambon. (*J. Pharm. Chim.*, 1901, xiii., [6], 57; through *Chem. Zeit. Rep.*, 1901, 40.)—The reagent consists of 3 or 4 grammes of pure dextrose dissolved in 100 grammes of hydrochloric acid. In a stoppered tube 7 or 8 c.c. of the reagent are shaken for two or three minutes with 15 c.c. of oil, the whole is heated till it begins to boil, shaken again, the tube closed and allowed to cool. The smallest trace of sesamé oil causes the liquid to appear pink, with a violet cast, which quickly changes to cherry-red. If 1 to 5 per cent. of sesamé are present, the colour develops in a few minutes; with 10 per cent. it shows immediately, and gradually becomes stronger. The reaction also succeeds with fatty acids. F. H. L.

Halphen's Reaction for Cotton Oil. Presence of Cotton Oil in American Lard. P. Soltsien. (*Zeits. öffentl. Chem.*, 1901, vii., 25; through *Chem. Zeit. Rep.*, 1901, 52.)—In consequence of Raikow's statements and his own investigations, the present author has now reverted to the use of amyl alcohol in this test. He adds 20 per cent. of a 1 per cent. solution of sulphur in carbon bisulphide to the oil or fat, and then introduces about one-half the total volume of amyl alcohol. Light at first hastens the appearance of the colour and then bleaches it. F. H. L.

The Characteristics of Lipase. J. H. Kastle and A. S. Lœvenhart. (*Amer. Chem. Journ.*, 1900, xxiv., 491-525.)—Ethyl butyrate is so rapidly hydrolyzed by lipase that it can be used to measure the activity of the enzyme.

In their experiments the authors prepared the extract of lipase by macerating 10, 20 or 50 grammes of the fresh pancreas of a pig with coarse white sand, extracting it with water or glycerin, and diluting to 100 c.c. In each case 4 c.c. of water, 0·1 c.c. of toluene (as a preservative), and 0·26 c.c. of ethyl butyrate were heated for five minutes at 40° C., and then, after the introduction of 1 c.c. of the extract, for a further forty minutes, after which the liquid was titrated with $\frac{N}{20}$ potassium hydroxide solution.

In comparison with the pancreas, the other tissues of a pig showed the following relative degrees of activity: pancreas, 1·0; liver, 2·93; kidney, 0·50; submaxillary gland, 0·36.

Comparative experiments with the extracts from the livers of other animals, in which the action of the enzyme was continued for fifteen minutes, gave the following amounts of hydrolysis: Pig, 8·66; sheep, 4·77; duck, 2·70; ox, 2·20; and chicken, 1·95 per cent.

On repeated filtration of the extracts, the lipase is almost completely removed from the liquid. It is more stable than is usually supposed, and the extract can be kept in a refrigerator for several days without losing its activity.

Ethereal salts are most rapidly hydrolysed by lipase at 40° C., whilst the enzyme is destroyed at 60°-70° C. Most of the common antiseptics are injurious, and, in particular, sodium fluoride, hydrofluoric acid, and acids in general.

The authors find that the velocity of the reaction is not proportional to the active mass of the ethereal salt, but is nearly so to the concentration of the enzyme. In the case of ordinary extracts the reaction is not complete, though it is practically so when very active extracts of the enzyme are used, or when the proportion of ethereal salt is very small. The co-efficient of the velocity is not constant, but decreases with the progress of the reaction.

By means of lipase, the authors have effected a synthesis of ethyl butyrate from butyric acid and alcohol, and from this conclude that the action of the enzyme is reversible. They consider that this discovery may throw light upon the question of the storage and utilization of fatty reserve material by plants. C. A. M.

Notes on the Examination of Beeswax. G. Buchner. (*Chem. Zeit.*, 1901, xxv., 21 and 37.)—Valuation of wax becomes more difficult when the Hübl constants are normal or nearly so, and the qualitative tests hint at impurities. In such cases the author's process (*Chem. Zeit.*, 1895, xix., 1422) is best employed to ascertain the "Buchner number"; Werder's recent method (*loc. cit.*) for the determination of neutral matter can also be recommended.

Occasionally the constants are abnormal, but the qualitative tests suggest purity. In general the limits laid down by Hübl and Allen (19 to 21, 73 to 76, 92 to 97, 3·6 to 3·8) are correct; sometimes, however, one obtains 17·5 to 21, 70 to 78, 87·5 to 99. The soft agreeably-smelling Ghedda wax (from British India and Bombay) often gives 5·33, 88·35, 93·68, 17·6; while Ahrens and Hett have obtained similar figures from humble-bee and cicada waxes. Ten per cent. of Ghedda wax added to a normal product would yield acid and ester values 18·53 and 76·3; 20 per cent. would show 17·06 and 77·6; 50 per cent., 12·65 and 81·67; yet each would be pure wax. African wax also gives abnormal constants. The author has received a sample of comb from Afrat, near Mogador. When this was purified by repeated melting it gave: specific gravity at 15° C., 0·961; melting-point, 64° to 65°; solidifying-point, 61°; iodine value, 11·65; and the average Hübl constants 19·92, 79·43, 99·35, 3·98. The stearic acid test showed a slight amorphous deposit in twenty-four hours, as almost all African waxes do; but no foreign matter could be detected. Chemically bleached wax of undoubted purity sometimes exhibits a high acid value, as the following figures indicate: 23·19 to 26·23, 69·00 to 73·93, 95·00 to 98·45, 2·70 to 3·20.

It is clear that the Hübl constants are no longer sufficient by themselves to enable the purity of beeswax to be judged. Qualitative tests must also be employed. The presence of 1 per cent. of stearic acid may correspond with the addition of 15 per cent. of "composition"; and often only 5 or 10 per cent. are mixed with the wax, for even this small amount of factitious matter represents a considerable annual profit to the manufacturer. F. H. L.

INORGANIC ANALYSIS.

Determination of Small Quantities of Silver in Sulphide Ores. A. Hollard. (*Rev. Phys. Chim.*, 1901, v., 55; through *Chem. Zeit. Rep.*, 1901, 61.)—One hundred grammes of the sample are dissolved in 10 c.c. of strong sulphuric acid and 300 c.c. of a mixture of 3 volumes of nitric acid of 36° B., and 1 volume of hydrochloric acid of 22° B. The whole is evaporated till sulphuric acid fumes have nearly disappeared, and taken up in a mixture of 75 c.c. of water, 5 c.c. of hydrochloric acid, and

20 c.c. of nitric acid of 36°. (If the ore contains much lead, the lead sulphate of the residue is extracted with sodium hydroxide of 12° Beaumé.) The insoluble matter is filtered off, washed, and dissolved in potassium cyanide. The solution is diluted to 250 c.c.—it should contain 2 per cent. of cyanide—and electrolyzed for a few hours with a current of 0·05 ampère. The silver is taken up in 50 c.c. of nitric acid and 50 c.c. of water, and determined by Volhard's method. F. H. L.

Volumetric Estimation of Mercuric Chloride in Aqueous Solution. A. Archetti. (*Boll. Chim. Farm.*, 1900, xxxix., 765; through *Chem. Zeit. Rep.*, 1901, 11.)—This process depends on the formation of "white precipitate" when mercuric chloride is treated with ammonia. A few drops of alcoholic phenolphthalein are added to the mercury solution, and decinormal ammonia is run in till the pink colour is permanent. Each c.c. of reagent corresponds to 0·271 gramme of corrosive sublimate. F. H. L.

A New Method of Separating Metals of the Platinum Group. Leidié. (*Journ. Pharm. Chim.*, 1901, xiii., 18-23.)—The industrial methods employed for the extraction of platinum and iridium from the ore leave residues which contain various rare metals. For the separation of these the author recommends the following method, based on the characteristics of their double alkali nitrites:

I. Elimination of Foreign Metals and Conversion of the Platinum Metals into Double Alkali Nitrites.—The residue is ignited in the air at a dull red heat, and reduced in hydrogen. It is next treated with water, then with hydrochloric acid, and again reduced in hydrogen. The metallic residue is mixed with twice its weight of sodium chloride, pulverized, and heated in a current of dry chlorine. The fixed and volatilized products are treated with a large excess of water acidulated with hydrochloric acid, in which process nearly the whole of the silver chloride is dissolved (by means of the sodium chloride present), whilst but little lead chloride is dissolved.

The solution is partially neutralized with sodium carbonate and brought nearly to the boiling-point. Sodium nitrite is now introduced little by little until the liquid becomes neutral to turmeric, at which point sodium carbonate is added until the precipitate no longer increases. The liquid is then boiled, left to cool, and filtered.

The sodium nitrite precipitates the iron as sesquioxide, and the gold in the metallic form. The sodium carbonate precipitates all such metals as lead, silver, zinc, tin, bismuth, copper, etc., originally present in the mineral or introduced in the treatment.

The metals of the platinum group remain in solution as double nitrites, having the following formulæ:

Nitrite of ruthenium and sodium	$Ru_2(NO_2)_6.4NaNO_2$.
,, of platinum ,, ...	$Pt(NO_2)_2.2NaNo_2$.
,, of palladium ,, ...	$Pd(NO_2)_2.2NaNO_2$.
,, of iridium	$Ir_2(NO_2)_6.6NaNO_2$.
,, of rhodium	$Rh_2(NO_2)_6.6NaNO_2$.

The osmium is present in the form of the double chloride, $OsCl_3.3NaCl$.

II. Separation of the Platinum Metals—Osmium, Ruthenium. — The nitrites mentioned above are not precipitated either by alkalies or alkali carbonates. Sodium hydroxide is added to their solution, and a current of chlorine passed through, whilst the volatile products are collected in water containing alcohol. The osmium and ruthenium salts are thus converted into the volatile peroxides OsO_4 and RuO_4. At the end of the operation the distillation flask is slightly heated to expel them completely with the steam. The alcohol in the receiver reduces them to the metallic state, and they are then separated from each other by Deville and Debray's method (*Ann. de Chim. et Phys.* [5], lvi., 476, 480).

Iridium, Rhodium.—The residual liquid is acidified with hydrochloric acid and boiled, and the metals reconverted into nitrites by the addition of sodium nitrite until the reaction is neutral. It is then cooled and saturated with ammonium chloride. This yields a precipitate of the double nitrites of iridium and rhodium with ammonium, which are insoluble in solutions of ammonium chloride. The precipitate is collected after twenty-four hours, and repeatedly treated with hot *aqua regia* to convert the nitrites into the respective chlorides $IrCl_4$ and Rh_2Cl_6. The *aqua regia* is expelled by evaporation, the residue taken up with cold chlorine-water, and the solution saturated with ammonium chloride.

The iridium is precipitated as the double chloride of ammonium and iridium. This is collected, dried, mixed with its own weight of sodium chloride, and heated in a current of chlorine at 440° to 450° C. By this means iridium is left as sodium iridium chloride, which is soluble in water, whilst any rhodium present is left as the anhydrous sesquichloride, which is insoluble in water.

The sodium iridium chloride is again converted into the ammonium salt, and the latter, when reduced by hydrogen, yields metallic iridium.

The filtrate from the iridium precipitate is evaporated to incipient crystallization. The crystals which then separate consist of a mixture of ammonium rhodium chloride ($Rh_2Cl_6.6NH_4Cl$) and ammonium chloride. The rhodium salt is converted into the double nitrite of sodium and rhodium, and this is precipitated as the ammonium salt. (If iridium were present, its corresponding salt, being a little more soluble, would be left in the mother liquid.) The ammonium rhodium nitrite is reconverted by hydrochloric acid into ammonium rhodium chloride, and metallic rhodium is obtained from this by reduction in hydrogen.

Platinum, Palladium.—The solution, after the removal of iridium and palladium, still contains double nitrites of platinum and palladium. It is evaporated to dryness after the addition of hydrochloric acid to convert the nitrites into chlorides. As the amount of sodium chloride present would interfere with the precipitation of the platinum, the residue is reduced in hydrogen. The metallic platinum and palladium, with possibly traces of iridium, are washed with water, dissolved in *aqua regia*, the solution evaporated, and the residue taken up with water. The liquid is placed in a flask, which it nearly fills, the air expelled by means of a current of carbon dioxide, and a current of nitrogen dioxide passed in as a reducing agent, and finally expelled by carbon dioxide.

The solution is now saturated with ammonium chloride and left for twenty-four hours, after which the ammonium platinum chloride is collected, recrystallized once or twice, and reduced in hydrogen to metallic platinum.

To the filtrate is added mercuric cyanide, which precipitates the palladium as palladious cyanide, $Pd(CN)_2$. This is ignited, and the residue dissolved in nitric acid. The nitrate is converted into palladious chloride, and then into the nitrite of potassium and palladium, which is converted into the double chloride of potassium and palladium, and crystallized. This salt is reduced at a red heat in hydrogen, and the residue, when cooled in a current of carbon dioxide and washed with water to remove the potassium chloride, leaves metallic palladium.

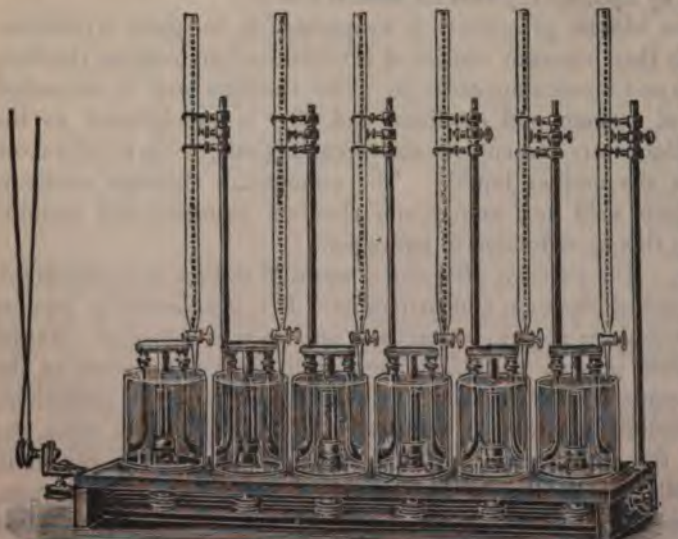
In the course of this treatment any iridium is converted into the hydrated sesquichloride, and is not precipitated. C. A. M.

Titration of Free Alkali in Presence of Hypochlorites, Chlorates, and Chromates. H. von Huber. (*Zeits. Electroch.*, 1901, vii., 396; through *Chem. Zeit. Rep.*, 1901, 32.)—The hypochlorite is reduced with normal sodium sulphite or sodium thiosulphate, the chromate precipitated with barium chloride, and the free alkali is titrated with normal hydrochloric acid, using methyl orange as indicator. If large quantities of alkali metal chromates are present, the barium chromate precipitate should be filtered off, and an aliquot portion of the liquid titrated.

F. H. L.

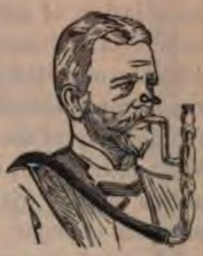
APPARATUS.

Apparatus for Parallel Titrations. A. Thilmany. (*Chem. Zeit.*, 1901, xxv., 115.)—The titrating vessels in this apparatus are glass cylinders with a central tube open at the bottom, and rising to the level of the outer part. Through these central tubes pass metal spindles with square heads, on which drop horizontal cross-pieces, and to the latter glass stirrers with metal caps are screwed. These agitators are bent outwards at their base so as almost to touch the walls of the glass vessels, while their upper parts revolve clear of the burette ends and taps. The central uprights are driven by gearing or belting contained in the box of the stand from any convenient prime motor. As all the stirrers travel at the same speed, the amount of agitation in all the vessels is



uniform; and the whole arrangement becomes specially suitable for comparative experiments in dye-testing, etc. The apparatus is protected as a "Gebrauchsmuster." F. H. L.

A Simple Respirator. A. Junghahn. (*Chem. Zeit.*, 1900, xxiv., 1138.)—As shown by the sketch, this apparatus is made of glass, with two automatic valves, the expired air escaping at the top, and that needed for inspiration being brought from behind the wearer through the rubber tube. A brass clip closes the nostrils. The device is equally suitable for laboratory or industrial purposes; it is stated not to impede the breathing, and, being all glass, is not itself corroded by acid vapours. The respirator is patented in Germany, and may be procured from Kaehler and Martini, Berlin, W. F. H. L.



REVIEWS.

Die Gährungsorganismen in der Theorie und Praxis der Alcoholgährungsgewerbe. By Albert Klöcker. Stuttgart: Max Waag. Price 9 marks.

The above work, which is dedicated to Dr. Hansen, of Copenhagen, will be welcomed by chemists and all others concerned with the alcoholic fermentation industries. It is divided into three chapters, the first of which gives a short but succinct account of the principal theories of fermentation which have been propounded since the introduction of the microscope. The second chapter, which contains a description of the apparatus used and the methods employed in a fermentation physiological laboratory, is a very important one from a practical point of view. The author, having acted as assistant at Carlsberg for a number of years, is able to give collectively the methods employed there—the fountain-head, as it were, of this kind of work; though those employed elsewhere are not neglected. Chapter III. contains a description of the chief micro-organisms met with in the alcoholic fermentation industries. Intermingled with the above subjects, which are indicated in mere outline, much information of an interesting and valuable nature is to be found, such as the variations, temporary or permanent, that can be artificially brought about in the *Saccharomycetes*, etc. The directions all through the book are given with such clearness and minuteness of detail as to render the work eminently adapted for self-instruction. It is copiously illustrated, the engravings of the micro-organisms being particularly fine. Not the least valuable feature of the book is the large and full synopsis of the literature of the subject given in the appendix, a noteworthy innovation being the addition of short abstracts to many of the references, which give the essential points contained in the treatises or papers mentioned. W. J. S.

A TEXT-BOOK OF URINE ANALYSIS, FOR STUDENTS AND PRACTITIONERS OF MEDICINE.
By JOHN H. LONG, M.S., Sc.D. Price $1·5 dollars.

The chief fault we should feel inclined to find with this book is that, while scarcely sufficiently comprehensive for the specialist, it is unnecessarily full for the practitioner. In most cases the various tests are clearly and concisely described, and the precautions necessary to avoid error are well explained. Unfortunately, however, one of the most important tests, from the point of view of the practitioner—viz., the coagulation of albumin by heat—is by no means well described. Filtration before boiling is not mentioned, nor the well-known expedients for contrasting the boiled with the unboiled part of the urine, and the best way of viewing the urine is also left to the, perhaps inexperienced, experimenter. Attention to these points greatly increases the delicacy of the test. The various tests for sugar are, on the whole, well described. We are, however, of opinion that Trommer's test might well have been omitted; for, unless applied with the greatest care, it is more apt to conceal than to reveal the presence of sugar. If it is to be used at all, the urine should, after the addition of the sulphate of copper, be filtered before boiling. The directions for the estimation of urea by hypobromite also leave something to be desired. All the apparatus described, with the exception of Huefner's original one, are only makeshifts, and in using them much time has to be wasted to allow the apparatus to fall to the temperature of the room. Also, why waste nearly a whole page in describing the Doremus apparatus, in which the author himself does not seem to believe?

The book is well printed, on good paper, and is commendably free from printer's errors. A. D.

SOCIETY OF PUBLIC ANALYSTS.

LETTER TO THE CORPORATION OF THE CITY OF LONDON.

[COPY.]

To the Corporation of the City of London.

THE CITY ANALYSTSHIP.

The Council of the Society of Public Analysts view with regret the terms offered by the Corporation of the City of London in respect of the appointment of City Analyst.

These terms, in the opinion of the Council, are very inadequate, especially in regard to analyses of water, and, in view of the increasing responsibilities thrown upon Public Analysts, are not such as to allow of the work being conscientiously and at the same time remuneratively performed.

The Council regret that a body so important as the Corporation of the City of London, instead of giving a man of the highest standing a remuneration worthy of his position and attainments, should have encouraged a system of competition among professional chemists to secure a prominent position without regard to the adequate remuneration of their services.

Your obedient servants,

EDWARD BEVAN } Hon. Secs.
ALFRED C. CHAPMAN }

LONDON, *April* 10, 1901.

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THE ANALYST.

MAY, 1901.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE monthly meeting of the Society was held on Wednesday evening, April 3, in the Chemical Society's Rooms, Burlington House.

In the absence of the President through indisposition, Mr. Otto Hehner occupied the chair.

The minutes of the previous meeting were read and confirmed.

Professor T. E. Thorpe, C.B., D.Sc., F.R.S., was elected an honorary member of the Society; and Messrs. H. Wippell Gadd and Albert H. Mitchell, B.Sc., were elected members.

A certificate of proposal for election to membership in favour of Mr. John Webster was read for the second time.

The following papers were read: "On the Maumené Test for Oils," by C. A. Mitchell, B.A.; "Some Arsenic Estimations relating to Malt Kilns," by Thomas Fairley; "On Gutzeit's Test for Arsenic," by F. C. J. Bird; and "The Aeration Test for Effluents," by S. Rideal, D.Sc.

COMPOSITION OF DUTCH BUTTER.

BY JOHN CLARK, PH.D.

(*Read at the Meeting, February* 5, 1901.)

DURING the last five years I have had occasion to analyse a large number of butters, all of which are certified by the importers as being derived from factories in Friesland, a province in the north of the Netherlands, in which, I understand, no margarine is manufactured. As each sample represents a shipment, I have thought it would be of some interest to publish the results which I have obtained, to show the extent to which the volatile acids in the fat may fall in butters the genuineness of which seems to be beyond question. All the results were obtained by Leffmann and Beam's alkali-glycerin process, which, in my opinion, is quite as accurate as the Reichert-Wollny method and more expeditious. A blank was made with the alkali-glycerin, and the distillation was carried to about one-fourth of the original volume. In most cases I employed a slight excess of sulphuric acid to decompose the soap, but I have found by numerous experiments that the same results are obtained when a considerable excess of phosphoric acid is used for this purpose. The volatile acids, after deducting the blank, are given in their equivalent of butyric acid:

Year.	Month.	Number of Samples.	Volatile Acids Per Cent.		
			Average.	Maximum.	Minimum.
1896	January	3	4·90	5·06	4·80
	February	5	5·17	5·52	4·80
	March	4	5·50	5·57	5·44
	April	1	5·54		
	May	2	5·27	5·36	5·18
	June	None			
	July	3	5·26	5·29	5·13
	August	4	5·37	5·50	5·24
	September	None			
	October	1	4·75		
	November	1	4·70		
	December	1	5·34		
1897	January	1	4·90		
	February	3	5·37	5·72	4·99
	March	1	4·96		
	April	None			
	May	2	5·50	5·72	5·28
	June	2	5·45	5·54	5·36
	July	None			
	August	1	5·44		
	September	None			
	October	1	5·00		
	November	2	4·73	4·90	4·57
	December	1	5·50		
1898	January	None			
	February	2	5·14	5·18	5·10
	March	None			
	April	None			
	May	None			
	June	5	5·55	5·90	5·01
	July	1	4·84		
	August	2	5·16	5·48	4·85
	September	3	4·96	5·08	4·81
	October	4	4·79	5·01	4·45
	November	9	4·88	5·14	4·62
	December	2	4·97	5·15	4·80
1899	January	2	5·15	5·20	5·10
	February	1	5·15		
	March	2	5·74	5·86	5·63
	April	3	5·88	6·06	5·80
	May	1	5·90		
	June	None			
	July	1	5·50		
	August	8	5·16	5·83	4·84
	September	3	5·15	5·20	5·05
	October	4	4·92	5·12	4·71
	November	6	4·77	5·04	4·43
	December	None			

			Volatile Acids Per Cent.		
Year.	Month.	Number of Samples.	Average.	Maximum.	Minimum.
1900	January	1	5·20		
	February	2	5·15	5·20	5·10
	March	3	5·65	5·96	5·40
	April	5	5·56	5·80	5·30
	May	None			
	June	3	5·45	5·74	5·30
	July	1	5·12		
	August	4	5·12	5·35	4·77
	September	16	4·80	5·17	4·45
	October	29	4·76	5·25	4·35
	November	34	4·67	5·18	4·20
	December	3	4·76	4·86	4·64

The above analyses indicate that the volatile acids in Friesland butter by this method are, on an average, 5·17 per cent. On seven occasions they fell below 4·5 per cent., but in several of these cases I have been able to prove the genuineness of the butter. In 1898 the sample giving 4·45 per cent. of volatile acids was reported as of doubtful purity, and for the satisfaction of the importers I was requested to send someone to visit the factory in which the butter was made. For this purpose I nominated Mr. B. A. van Ketel, analyst in Amsterdam, who forwarded to me a sample of butter, which he certified was churned in his presence, and a portion of the cream from which it was made. The analyses of the fat of each of these gave the following results:

	Butter Fat.	Cream Fat.
Volatile acids per cent.	4·42	4·45

In October of last year two samples of butter from different factories showing 4·35 per cent. of volatile acids were also reported as of doubtful purity, and to satisfy the importers I again asked Mr. van Ketel to visit these factories, and send me samples of the butter and of the cream. On analysis the fats of these gave the following results:

	No. 1. Butter.	No. 1. Cream.	No. 2. Butter.	No. 2. Cream.
Volatile acids per cent.	4·30	4·35	4·33	4·33

In November two of the butters gave only 4·2 and 4·22 per cent. of volatile acids, and although I had no opportunity in these cases of examining certified samples from the churn, I received a letter from Dr. K. H. M. v. d. Lande, Director of the Government Agricultural Station at Hoorn, stating that on November 10 he had analysed a sample of butter from the same factory, taken from the churn in presence of witnesses, and that he found it to contain 4·19 per cent. of volatile acid, calculated as butyric acid.

The above results indicate that the poverty in volatile acid in these cases is not due to adulteration, but to natural causes, and this seems to take place mainly from about the middle of September till the middle of November, and is said to be due to the exposure of the cattle to the cold, combined with indifferent feeding, as the proportion of volatile acid gradually increases after the cattle are housed.

DISCUSSION.

Mr. HEHNER said that it was very difficult to discuss such a paper as this in the absence of the author. He would have liked to ask what the author meant when he spoke of volatile acids. He gathered that the process used was practically the Wollny process, excepting that the saponification was carried out according to Leffmann's method, with sodium hydroxide and glycerin. It was, however, practically impossible to distil off completely the volatile fatty acids, and as no correction appeared to have been made for that portion of the acids which did not distil over, the figures would be simply the Wollny figures, multiplied by a factor for conversion into percentages of volatile fatty acids. Unfortunately, the results of such processes were ordinarily recorded, not in percentages of volatile fatty acids, but in c.c. of decinormal alkali neutralized by a given quantity of butter. It would have been much more satisfactory if a system of the kind followed in this paper had been adopted from the first: as it was, it was almost impossible to form any opinion on the figures as they stood. For many years past the Dutch butter importers had been up in arms every November against analysts in England who condemned samples representing consignments of Dutch butter, which were said to be perfectly genuine. There was no doubt that this state of things was in a great measure due to the fact that in Holland the calving of the cows was managed in a preposterously bad manner. The cows somehow or other were allowed to get dry all at the same time, and consequently all at the same time to reach a condition in which the fat of their milk was quite abnormal in composition. This occurred at about the end of October or the beginning of November, just at the same time at which the change took place from the open air to stall feeding, a change which deeply affected the composition of the butter-fat. Taking these two circumstances together, it might very well happen, apart from any question of fraud, that the butter might analyse abnormally; but no such fluctuations would occur if the Dutch farmers chose to arrange that the calving of their cows was fairly divided over the year, as was the case in any country where farming was reasonably conducted, and if they stabled their cows rather earlier than hitherto. He understood that this matter was now being discussed by the more intelligent of the Dutch farmers themselves. As a result of this unfortunate management, and the difficulties which arose in consequence, Dutch butter had become discredited to such an extent that the Belgian Government now enacted that no butter should be exported from Belgium which gave a Reichert-Wollny figure lower than 25.

Mr. BEVAN said it seemed quite clear that the process which Dr. Clark had used was analogous to the Reichert-Wollny process, and he thought it quite right to assume, therefore, that the figures were comparable with the Reichert-Wollny figures expressed in terms of c.c. of decinormal alkali.

Dr. DYER said that he would have liked to ask Dr. Clark to restate these figures in what were now the usual terms. He thought that Mr. Hehner's view was probably correct as to the curious arbitrary method of statement which was used in the Reichert process; but that method of statement was now so universally adopted that it did not seem desirable to change it. He did not agree that it would be better to state the results in terms of the volatile acids, for, in speaking of percentages of volatile acids, the calculation was made on the assumption (which of course was also arbitrary) that the volatile fatty acids all consisted of butyric acid, which was not the case; whereas the Reichert figure was at least a statement of fact, viz., that a certain quantity of butter yielded under certain circumstances a quantity of volatile fatty acids represented by a certain quantity of standard alkali.

(The following reply to some of the points in the discussion has been received from Dr. Clark.—Ed.)

The "volatile acids" are stated in terms of butyric acid, and are calculated from the Reichert-Wollny figures. They represent the volatile acids distilled over, *not* the total volatile or butyric acid. This is the manner in which I always report my butter analyses, and is one more easily understood by a court. The process employed is analogous to the Reichert-Wollny process, and the results given can be converted into the Reichert-Wollny figures, expressed in terms of c.c. of decinormal alkali, by multiplying the volatile acids by 5·682. In this way it will be found that 4·4 per cent. of butyric or volatile acid corresponds to the Reichert-Wollny figure 25.

THE PHYSICAL STATE IN WHICH FAT EXISTS IN CREAM.

By H. Droop Richmond, F.I.C., and Sylvester Oliffe Richmond.

(*Read at the Meeting, February* 5, 1901.)

One of us has already shown that there is a distinct change of density of cream at about the melting-point of the fat ("Dairy Chemistry," Appendix A., pp. 338-341); it was further shown that separated milk exhibits no such change, and that the expansion of the fat in cream agrees with the expansion of butter-fat, assuming that it is liquid above its melting-point and solid below that. This is true of cream that has been kept for some hours at a low temperature, and does not hold with cream that has been heated and rapidly cooled; the density results point to the assumption that the solidification of the fat is a process which goes on only slowly.

As the density gives only indirect evidence, and as it has been concluded by several authorities that the fat in milk is always liquid, we have thought it desirable to study the question in another way.

If a body is receiving heat from any source, the rate of rise of temperature will depend (*a*) on the difference of temperature, and (*b*) on the specific heat-capacity; if at any point a change involving absorption or evolution of heat takes place, the curve of temperature will show discontinuity at that point, and if, in addition, the change causes an alteration of specific heat-capacity, there will not only be discontinuity, but also a change of curvature at that point.

As it is extremely unlikely that the specific heat capacities of solid and liquid fat are the same, we should expect to find a change of the rate of heating at the melting-point of fat.

The method adopted was to place about 20 c.c. of cream in a tube in which were a thermometer reading directly to $\frac{1}{10}$° C., and by estimation to $\frac{1}{100}$° C., and a stirrer;

TABLE I.

SERIES I.—WELL-COOLED CREAM.

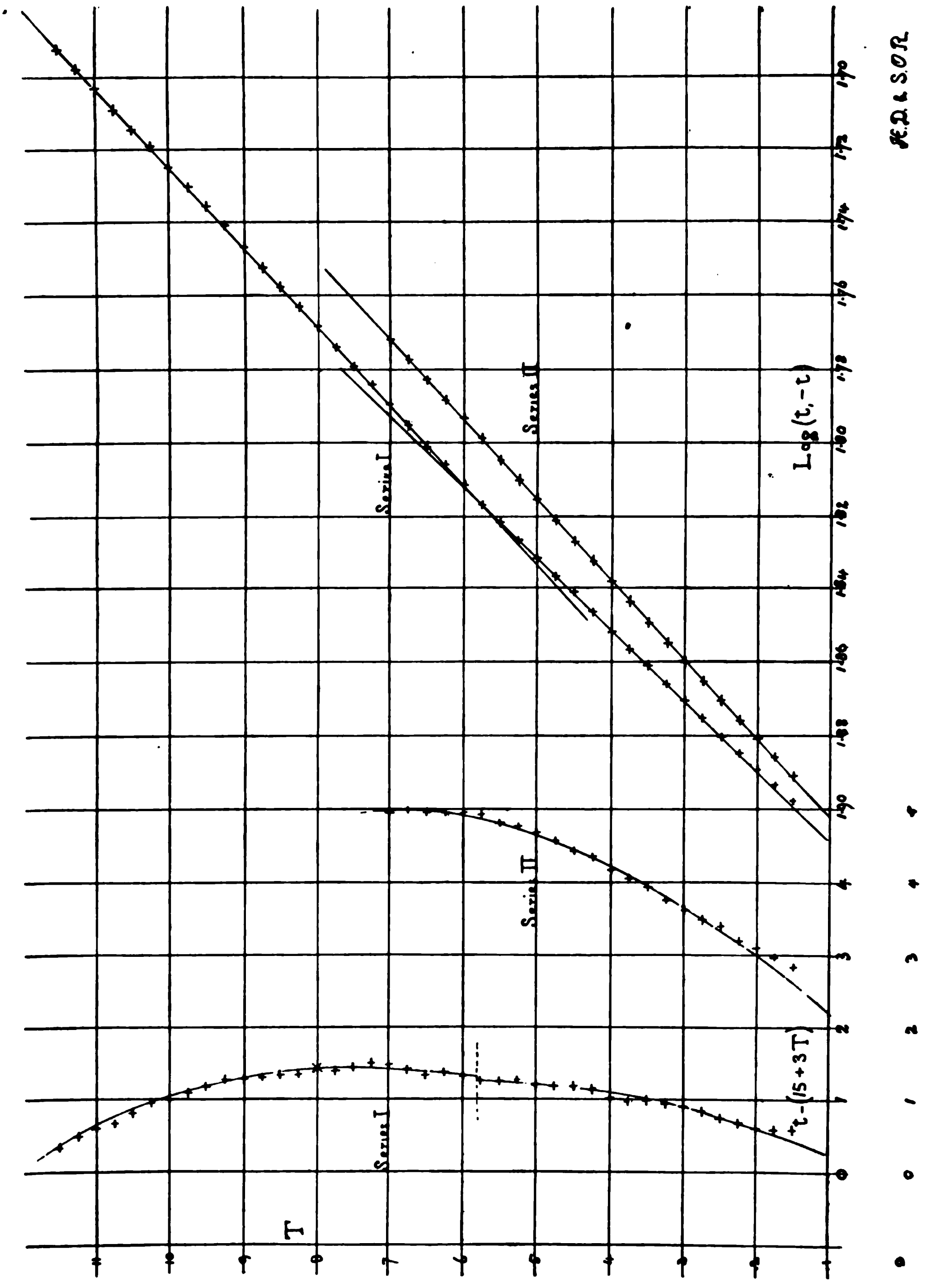
A.	B.	C.	D.	E.	F.
0·25	17·28	82·80	1·9180		
0·5	17·95	82·13	1·9145	0·0035	0·0081
0·75	18·70	81·38	1·9105	0·0040	0·0092
1·0	19·50	80·58	1·9062	0·0043	0·0099
1·25	20·25	79·83	1·9022	0·0040	0·0093
1·5	21·10	78·98	1·8975	0·0047	0·0107
1·75	21·85	78·23	1·8934	0·0041	0·0095
2·0	22·62	77·46	1·8890	0·0044	0·0099
2·25	23·45	76·63	1·8844	0·0046	0·0108
2·5	24·25	75·83	1·8799	0·0045	0·0106
2·75	25·10	74·98	1·8750	0·0049	0·0112
3·0	25·92	74·16	1·8702	0·0048	0·0110
3·25	26·70	73·38	1·8656	0·0046	0·0106
3·5	27·48	72·60	1·8609	0·0047	0·0107
3·75	28·22	71·86	1·8565	0·0044	0·0102
4·0	29·02	71·06	1·8517	0·0048	0·0114
4·25	29·90	70·18	1·8482	0·0053	0·0124
4·5	30·70	69·38	1·8412	0·0050	0·0115
4·75	31·43	68·65	1·8366	0·0046	0·0106
5·0	32·21	67·87	1·8317	0·0049	0·0114
5·25	33·03	67·05	1·8264	0·0053	0·0122
5·5	33·76	66·32	1·8216	0·0048	0·0110
5·75	34·49	65·59	1·8168	0·0048	0·0111
6·0	35·32	64·76	1·8113	0·0055	0·0127
6·25	36·14	63·94	1·8058	0·0055	0·0127
6·5	36·84 (?)	63·24	1·8010	0·0048	0·0110 (?
6·75	37·68	62·40	1·7952	0·0058	0·0140 (?
7·0	38·49	61·59	1·7895	0·0057	0·0131
7·25	39·26	60·82	1·7840	0·0055	0·0126
7·5	39·95	60·13	1·7791	0·0049	0·0114
7·75	40·65	59·43	1·7740	0·0051	0·0117
8·0	41·43	58·65	1·7683	0·0057	0·0132
8·25	42·10	57·98	1·7633	0·0050	0·0115
8·5	42·84	57·24	1·7577	0·0056	0·0128
8·75	43·56	56·52	1·7522	0·0055	0·0127
9·0	44·29	55·79	1·7466	0·0056	0·0132
9·25	45·03	55·05	1·7408	0·0058	0·0133
9·5	45·69	54·39	1·7355	0·0053	0·0121
9·75	46·35	53·73	1·7302	0·0053	0·0122
10·0	47·00	53·08	1·7249	0·0053	0·0121
10·25	47·70	52·38	1·7192	0·0057	0·0133
10·5	48·31	51·77	1·7141	0·0051	0·0117
10·75	48·93	51·15	1·7088	0·0053	0·0120
11·0	49·62	50·46	1·7029	0·0059	0·0136
11·25	50·25	49·83	1·6975	0·0057	0·0126
11·5	50·85	49·23	1·6923	0·0052	0·0121

SERIES II.—HEATED CREAM.

A.	B.	C.	D.	E.	F.
0·25	17·95	82·13	1·9145	0·0045	0·0104
0·5	18·80	81·28	1·9100	0·0049	0·0111
0·75	19·70	80·38	1·9051	0·0047	0·0110
1·0	20·58	79·50	1·9004	0·0048	0·0110
1·25	21·45	78·63	1·8956	0·0049	0·0112
1·5	22·33	77·75	1·8907	0·0050	0·0115
1·75	23·22	76·86	1·8857	0·0050	0·0115
2·0	24·10	75·98	1·8807	0·0049	0·0113
2·25	24·95	75·13	1·8758	0·0055	0·0127
2·5	25·90	74·18	1·8703	0·0050	0·0115
2·75	26·75	73·33	1·8653	0·0053	0·0121
3·0	27·63	72·45	1·8600	0·0053	0·0125
3·25	28·52	71·56	1·8547	0·0056	0·0131
3·5	29·45	70·63	1·8491	0·0055	0·0122
3·75	30·31	69·77	1·8436	0·0054	0·0127
4·0	31·19	68·89	1·8382	0·0058	0·0134
4·25	32·11	67·97	1·8324	0·0054	0·0121
4·5	32·93	67·15	1·8270	0·0058	0·0135
4·75	33·83	66·25	1·8212	0·0057	0·0130
5·0	34·69	65·39	1·8155	0·0055	0·0128
5·25	35·52	64·56	1·8100	0·0055	0·0125
5·5	36·32	63·76	1·8045	0·0058	0·0136
5·75	37·18	62·90	1·7987	0·0054	0·0123
6·0	37·95	62·13	1·7933	0·0052	0·0120
6·25	38·69	61·39	1·7881	0·0055	0·0128
6·5	39·46	60·62	1·7826	0·0055	0·0128
6·75	40·23	59·85	1·7771	0·0053	0·0121
7·0	40·95	59·13	1·7718		

this tube was fixed inside a second wider tube immersed in boiling water, the temperature of which was noted; the cream was kept constantly stirred, and readings of the thermometer were made every fifteen seconds.

The chief source of error is due to the lag of the thermometer, which will, of course, decrease as the rate of rise of temperature becomes less. Other sources of error are imperfect stirring, which was guarded against by as vigorous a motion as possible, and the difficulty of estimating the temperature of the exposed thread of mercury, as this was not constant throughout. The last source of error is not so serious, as the total correction up to 40° is small, and a fairly large error in estimating the temperature of the bath is not important. The maximum error does not, we think, exceed 0·1° C.


Log(t₁-t)
1.70
1.72
1.74
1.76
1.78
1.80
1.82
1.84
1.86
1.88
1.90
Series I
Series II
Series II
Series I
t-(15+3T)
0
1
2
3
4
T
1
2
3
4
5
6
7
8
9
10
11
H.D. & S.O.R.

Two series of estimations were made: Series 1 with cream that had been kept in an ice-safe for eighteen hours, and Series 2 with cream that had been heated to 50° C. and cooled down to 15° as rapidly as possible; the cooling occupied about a quarter of an hour.

In Table 1 are given: *A*, the time of reading; *B*, the corrected temperatures; *C*, the differences between the temperature of the cream and that of the water (100·08° C.); *D*, the logarithms of the figures in column *C*; *E* the differentials of the figures in column *D*; and *F*, the differences between consecutive figures in column *B*, divided by the figures in column *C*.

We have plotted out on the plate* the figures in columns *B* and *D* against those in column *A*.

An inspection of the curves shows the following:

1. The curve of temperatures is continuous with the heated cream, but is discontinuous with the cooled cream; the point where discontinuity takes place is about 33° C.

2. The curve of the logarithms is nearly a straight line with the heated cream, and shows no well-marked break (if any at all); with cooled cream, on the other hand, the curve can only be expressed by two straight lines, with a well-marked break at about 34° C.

It is significant, though, considering the magnitude of the sources of error, not absolutely conclusive, that the rate of rise of temperature of cooled cream falls off about 33°-34°, as shown by the smaller figures; at these temperatures, in columns *E* and *F*, no falling off is noticed with heated cream.

We have also calculated the temperature (Table II.) at which the cream should be for each quarter-minute by a formula of the type $\log.\ (t_1 - t) = \mathrm{K} + c\mathrm{T}$, where T = time, t_1 the temperature of the water, and t the temperature of the cream. We have had to use two formulæ in Series 1, (*a*) for the lower branch and (*b*) for the upper branch; the constants $c = 0{\cdot}01948$ and $0{\cdot}02172$ respectively. One formula suffices for Series 2, with a constant $c = 0{\cdot}02188$. The results for the first 2½ minutes have been neglected in each case, as the introduction of the tube cooled the water somewhat, and the difference of temperature $t_1 - t$ was not steady till then.

It is seen in Series 1 that the point 34·49° lies equally well on both curves, and the values below all agree with the figures calculated by the lower formula within 0·10°; values above show a wider divergence. The values above agree equally well with the figures calculated by the upper formula, and values below show a very marked divergence from the figures calculated by this formula.

In Series 2 the agreement is almost as good throughout the curve, but it is to be remarked that the differences are all plus below 33, and all minus above; this, we think, clearly indicates a very slight break in the curve at this point, very much less than in Series 1, and not noticeable on plotting the curves, but still quite marked.

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* As the scale of plotting necessary for reproduction would fail to show clearly the discontinuity of curvature in Series 1 if temperatures (t) and time (T) were plotted out against each other, the curves have been opened out by the substitution of the expression $t - (15 + 3T)$ for t. This does not change the character of the curves, but is equivalent to plotting them with obtuse-angled instead of rectangular co-ordinates.

TABLE II.

Series I.						Series II.		
Time.	Temp.	Temp. (Calc. *a*).	Diff.	Temp. (Calc. *b*).	Diff.	Temp.	Temp. (Calc.).	Diff.
2·5	24·25	24·17	-0·08			25·90	25·87	-0·03
2·75	25·10	25·03	-0·07			26·75	26·80	+0·05
3·0	25·92	25·85	-0·07			27·63	27·70	+0·07
3·25	26·70	26·69	-0·01			28·52	28·61	+0·09
3·5	27·48	27·51	+0·03			29·45	29·51	+0·06
3·75	28·22	28·31	+0·09			30·31	30·39	+0·08
4·0	29·02	29·11	+0·09			31·19	31·26	+0·07
4·25	29·90	29·90	—			32·11	32·13	+0·02
4·5	30·70	30·69	-0·01			32·93	32·95	+0·02
4·75	31·43	31·46	+0·03	31·08	-0·35	33·83	33·81	-0·02
5·0	32·21	32·25	+0·04	31·94	-0·27	34·69	34·65	-0·04
5·25	33·03	32·99	-0·04	32·78	-0·25	35·52	35·46	-0·06
5·5	33·76	33·73	-0·03	33·63	-0·13	36·32	36·27	-0·05
5·75	34·49*	34·47	-0·02	34·45	-0·04	37·18	37·07	-0·11
6·0	35·32	35·21	-0·11	35·26	-0·06	37·95	37·86	-0·09
6·25	36·14	35·92	-0·22	36·08	-0·06	38·69	38·63	-0·06
6·5	36·84 (?)	36·64	-0·20	36·86	+0·02	39·46	39·40	-0·06
6·75	37·68			37·65	-0·03	40·23	40·17	-0·06
7·0	38·49			38·42	-0·07	40·95	40·92	-0·03
7·25	39·26			39·20	-0·06			
7·5	39·95			39·96	+0·01			
7·75	40·65			40·68	+0·03			
8·0	41·43			41·44	+0·01			
8·25	42·10			42·16	+0·06			
8·5	42·84			42·88	+0·04			
8·75	43·56			43·60	+0·04			
9·0	44·29			44·30	+0·01			
9·25	45·03			44·99	-0·04			
9·5	45·69			45·66	-0·03			
9·75	46·35			46·35	—			
10·0	47·00			47·01	+0·01			
10·25	47·70			47·67	-0·03			
10·5	48·31			48·33	+0·02			
10·75	48·93			48·97	+0·04			
11·0	49·62			49·60	-0·02			
11·25	50·25			50·24	-0·01			
11·5	50·85			50·86	+0·01			

Considering the results, we conclude that:

1. With well-cooled cream a change in specific heat-capacity and a slight but distinct absorption of heat occurs at about 34°.

2. With heated cream rapidly (during ¼ hour) cooled a very slight change occurs at this point.

3. The only possible change which would produce these results is the melting of the fat.

4. As a final conclusion, the fat in well-cooled cream is solid; the fat in rapidly-cooled cream is still for the most part liquid or superfused, though there are distinct

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* This point lies on both curves.

indications that a slight solidification has taken place in a quarter of an hour, and therefore the process of solidification is a slow one. The melting is practically instantaneous.

These conclusions are in complete accord with the density experiments previously referred to.

NOTES ON THE PROXIMATE ANALYSIS OF CLOVES.

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By A. McGill, B.A., Inland Revenue, Canada.

(Read at the Meeting, February 5, 1901.)

Recorded analyses of genuine cloves (flower-buds of *Caryophyllustus aromaticus*) are not numerous. The following notes may therefore possess interest.

All the samples were procured direct from importers, and were ground by myself to a uniform degree of fineness. The classifications as Penang, Amboyna, and Zanzibar are based on the statements of the importers, but the grading in each class is based upon my own observation of the following physical characters: Colour, size, plumpness, retention of corolla, and freedom from stems. The best samples I have graded as first quality, the worse as second quality, and in a few cases, being unable with certainty to assign to either of these grades, I have given the number 1½. This grading was made quite independently of the results of analysis, and it is interesting to observe how the physical characters of the whole clove give a suggestion as to its chemical character.

The most important constituent of cloves is the volatile or "essential" oil, and I have sought to modify the ordinary methods of working in such a way as to determine this component with the greatest degree of accuracy attainable by a practical, commercial process of assay.

The subjoined table gives percentage results obtained:

PENANG CLOVES.

No.	Moisture.	Total Volatile Matter.	Volatile Oil.	Total Extractive Matter.	Fixed Oil.	Quality.	Remarks.
1	5·0	21·2	16·2	28·2	12·0	1	
2	7·4	24·0	16·6	27·0	10·4	1	
3	5·8	20·7	14·9	24·4	9·5	2	Off colour, and resembled Zanzibar samples.
4	5·2	22·4	17·2	27·1	9·9	1	
5	6·9	21·7	14·8	26·5	11·7	1	
6	6·9	23·2	16·3	28·1	11·8	1½	A few stems.
7	7·1	24·3	17·2	28·2	11·0	1	
8	5·5	21·6	16·1	26·2	10·1	1	
Mean	6·2	22·4	16·2	27·0	10·8	—	Vol. oil : fixed oil :: 100 : 67.
AMBOYNA CLOVES.							
9	6·7	25·9	19·2	29·2	10·0	1	
10	5·5	23·5	18·0	26·7	8·7	1	
11	6·0	24·3	18·3	26·5	8·2	1	
Mean	6·1	24·6	18·5	27·5	9·0	—	Vol. oil : fixed oil :: 100 : 49.

ZANZIBAR CLOVES.

No.	Moisture.	Total Volatile Matter.	Volatile Oil.	Total Extractive Matter.	Fixed Oil.	Quality.	Remarks.
12	5·1	22·4	17·3	25·3	8·0	1	
13	6·0	24·3	18·3	28·1	9·8	1	
14	5·4	22·9	17·5	27·0	9·5	1	
15	5·7	18·4	12·7	21·3	8·6	2	Stems and shrunken buds.
16	6·3	22·9	16·6	26·3	9·7	1	
17	5·7	22·1	16·4	26·1	9·7	1	
18	6·5	18·6	12·1	22·3	10·2	2	Like No. 15.
19	6·5	21·0	14·5	25·2	10·7	2	,, ,,
20	6·2	23·6	17·4	27·8	10·4	1	
21	4·6	22·1	17·5	—	—	1	
22	4·1	21·4	17·3	—	—	1	
24	6·7	20·8	14·1	—	—	1½	Stems.
26	5·8	21·1	15·3	—	—	1½	,,
Mean	5·7	21·7	16·0	25·5	9·6	—	Vol. oil : fixed oil :: 100 : 59.
Mean, omitting 18	5·7	22·2	16·5	26·8	10·3	—	Vol. oil : fixed oil :: 100 : 62.
UNCLASSIFIED CLOVES.							
23	7·5	17·4	9·9	—	—	2	West Coast cloves.
25	6·9	21·9	15·0	—	—	1	
27	4·4	16·7	12·3	—	—	2	
28	7·5	13·4	5·9	—	—	—	Clove stems.

As in all proximate work of this kind much depends upon details in the process employed, it is necessary to give the following particulars:

Recorded analyses of genuine cloves give water from 2·90 to 16·39 per cent. (König, "Zusammensetzung," etc., p. 744). It will be noted that I have not found so large a range as this, my extremes being 4·1 and 7·5 per cent.

On exposing samples of powdered cloves, *in vacuo*, over sulphuric acid, I found that loss of weight continued to occur for many days; but that the rate decreased very rapidly after the first period of twenty-four hours, while the acid continued to grow deeper in colour till it became quite black from separated carbon. The subjoined table gives results obtained with six portions of 2 grammes of the same sample of cloves:

Hours in Desiccator.	Percentage Loss of Weight.							Remarks.
	a.	b.	c.	d.	e.	f.	Means.	
24	5·1	5·2	—	—	—	—	5·15	Acid scarcely discoloured.
48	5·4	5·7	5·5	4·6	5·1	4·8	5·15	,, distinctly brown.
72	5·8	6·1	5·8	5·1	5·2	5·2	5·55	,, more distinctly brown.
96	—	—	7·3	6·1	7·1	6·3	6·70	,, very brown.
120	7·9	8·2	—	—	—	—	8·05	,, very dark brown.
144	8·6	9·2	—	—	—	—	8·90	,, ,, ,,
	Desiccator 1.		Desiccator 2.					

The change in the rate of loss of weight, together with the fact that the volatile matter lost during the first twenty-four hours does not blacken the acid, leads me to conclude: (1) That water vapour only goes off during this period; (2) that loss of weight after the first twenty-four hours is essentially due to volatile oil. I found that at the end of sixteen days the total loss amounted to 11·45 per cent. The same sample of cloves lost 3·65 per cent. in twenty-four hours over sulphuric acid at ordinary pressure, and 4·72 per cent. in fifty-four hours, with only a slight discoloration of the acid. The advantage of drying *in vacuo* is chiefly the saving in time; but it is possible that the vapour tensions of water and essential oil of cloves are affected in such a way as to more sharply mark the point at which the oil comes off in notable quantity. In six samples treated in two desiccators of similar form, placed side by side, one being under atmospheric pressure, the other under 60 millimetres mercury, the following results were obtained for twenty-four hours:

	1.	2.	3.	4.	5.	6.
Reduced pressure ...	5·65	6·25	5·70	6·50	6·45	6·15
Ordinary pressure ...	4·65	5·15	4·65	4·85	4·95	5·20
Difference ...	1·00	1·10	1·05	1·65	1·50	0·95

Moisture, as given in the tabulated analyses, has been determined by exposure for twenty-four hours, over sulphuric acid under a reduced pressure of about 60 millimetres mercury.

Total volatile matter has been determined by exposure in a current of dry air at 98° C. for twenty-four hours. I find that a slight increase of volatile matter is obtained by previous treatment of the sample (in the capsule) with petroleum spirit. Two grammes of the sample are allowed to stand, at the ordinary temperature, after addition of 25 c.c. ether, until this ether is evaporated. It is then dried in the water-oven at 98° C. as described. Three samples gave results as below:

	1.	2.	3.
After ether treatment ...	20·72	21·52	9·47
Without ether	20·08	20·88	9·46
Difference	0·64	0·64	0·01

On repeating the treatment a further loss of weight occurred as follows: (1) 3·14, (2) 1·90 per cent. A third treatment yielded: (1) 0·46, (2) 0·30 per cent.; so that from 2 to 3 per cent. more volatile matter is obtained by a second treatment with ether. The results tabulated have been obtained by a single treatment as described.

The total extractive matter is obtained by Soxhlet extraction to constant weight. A check upon the volatile oil has been attempted by evaporating the solvent from this extractive, and drying, first at a low temperature and then at 100° C.; but it has been found impracticable to obtain constant weight, at either of these points, within 2 or 3 per cent. of error.

It is to be noted that, so far as the samples which would grade No. 1, on the basis of their physical properties, are concerned, the maximum and minimum percentages of volatile oil are as follows:

	Max.	Min.	Mean.
Penang	17·2	14·8	16·2
Amboyna	19·2	18·0	18·5
Zanzibar	18·3	16·4	16·0
Mean, for a mixture of equal weights of the three kinds	18·2	16·4	16·9
Values found by C. Richardson	18·89	10·23	15·63

(Washington, Department of Agriculture, Bull. 13.)

The lowest value which I have found for any whole clove claiming to be genuine is 12·1 per cent. in No. 18. I am certain that neither this sample, nor Numbers 15, 19, 24, 23, or 27 would be considered other than low grade by dealers who are in the habit of handling cloves.

I am therefore of opinion that no sample of cloves claiming to be of good quality should yield less than 14 per cent. of volatile oil, when assayed by the method which I have described.

If total volatile matter is determined by treatment with petroleum spirit before drying at 100° C., this treatment being repeated, the minimum volatile oil found should be 16 per cent.

DISCUSSION.

Mr. CHATTAWAY said that the author's statement as to the percentage of oil in cloves was well borne out by work which he had himself done on this subject, though in a somewhat different way. On the Continent there were to be found plenty of synthetic volatile oils, the use of which for such purposes must certainly, he thought, be regarded as reprehensible, though he believed that some of them found their way into well-known preparations.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

A Rapid Method for the Detection of "Aniline Orange" in Milk. Hermann C. Lythgoe. (*Journ. Amer. Chem. Soc.*, xxii., 813.)—The test may conveniently be carried out on the portion of the milk which is to be tested for formic aldehyde. Fifteen c.c. of the milk are placed in a porcelain basin, an equal quantity of hydrochloric acid (specific gravity 1·20) is added, and the basin gently shaken. If the milk contains aniline orange the curd will be coloured pink, otherwise it will be white or yellowish. To test for formic aldehyde the same solution is boiled after the addition of a drop of a solution of ferric chloride, when a purple colour will appear if formaldehyde is present. Hydrochloric acid, to which ferric chloride solution (5 c.c. of a 10 per cent. solution for 2 litres) has been added, may also be used for the two tests. The presence of both substances has no effect on either reaction.

Of the artificially-coloured samples of milk examined by the author during 1900, 48 per cent. contained aniline orange, and its use is on the increase. A. G. L.

The Determination of Lactose in Milk. A. Scheibe. (*Zeit. anal. Chem.*, 1901, xl., 1-14.)—After discussing the various methods of estimation, the author recommends the following processes:

Gravimetric Determination.—Twenty-five c.c. of the milk are diluted with 400 c.c. of water and treated with 10 c.c. of the copper sulphate portion of Fehling's solution (69·28 grammes per litre). After the further addition of 3·5 to 4 c.c. of normal sodium hydroxide solution and 20 c.c. of a cold saturated solution of sodium fluoride, the liquid is allowed to stand for thirty minutes, and then made up to 500 c.c. and filtered; 100 c.c. of the filtrate are boiled for six minutes in a deep porcelain dish with 50 c.c. of Fehling's solution, the cuprous oxide reduced in hydrogen, and the corresponding lactose obtained from Soxhlet's table (ANALYST, xx., 228).

Polarimetric Determination.—To 75 c.c. of the milk are added 7·5 c.c. of 20 per cent. sulphuric acid and 7·5 c.c. of a solution of mercuric iodide, prepared by dissolving 40 grammes of potassium iodide in 200 c.c. of water, shaking the solution with 55 grammes of mercuric iodide, diluting to 500, and filtering the supernatant liquid.

The mixture is next diluted to 100 c.c., and filtered, and the filtrate polarized in a 4 d.c.m. tube at 17·5° C. With a Schmidt and Hänsch half-shadow apparatus provided with a double quartz compensation-wedge 1 degree corresponds to 0·16428 gramme of lactose in 100 c.c. When using a polarization apparatus with circular degrees and sodium light, the reading should be made at 20° C., and 0·4759 gramme of lactose per 10 c.c. calculated for each degree.

In correcting the error due to the volume of the precipitate the result may be multiplied by 0·94 in the case of whole milk (2·8 to 4·7 per cent. of fat), and by 0·97 in the case of skim milk. In making the correction for cream or colostrum it is essential to determine the volume of the precipitate in the following manner:

(*a*) Ten grammes of pure lactose are dissolved in 75 c.c. of water, and the solution mixed with 7·5 c.c. of 20 per cent. sulphuric acid and 7·5 of the mercuric iodide solution, diluted to 100 c.c., filtered and polarized. M_1 in the equation represents the amount of lactose in grammes thus found.

(*b*) Ten grammes of lactose are dissolved in 75 c.c. of milk with the aid of heat, the cold solution treated with 0·5 c.c. of concentrated ammonium hydroxide, and after ten minutes mixed with 7·5 c.c. of sulphuric acid (20 per cent.), and 7·5 of the mercuric iodide solution, diluted to 100 c.c., filtered and polarized. The difference between this reading and that obtained with 75 c.c. of the milk alone corresponds with the apparent amount of lactose added to the milk, or M_2 in the equation.

(*c*) The volume of the precipitate is calculated in the following manner: The volume of the liquid + that of the precipitate ($=NV$) $=100$ c.c. This volume corresponds to M_2; the volume of the liquid $=100-NV$, which corresponds to M_1, or

$$100 : M_2 = (100 - NV) : M_1$$
$$NV = \frac{100\ M_2 - 100\ M_1}{M_2}.$$

(*d*) The apparent amount of lactose in the milk multiplied by $\frac{100-NV}{100}$ gives the actual amount.

C. A. M.

The Detection of Cocoanut Oil in Butter. **J. Wauters.** (*Bull. de l'Ass. belg.*, 1901, xv., 25-28.)—In the determination of the Reichert-Meissl value a considerable proportion of insoluble fatty acids distil over with the volatile acids (*cf.* ANALYST, xxvi., 71).

The author has made general experiments in this direction, but has abandoned them in favour of the following method of distillation, in which the high temperature due to the concentration of the liquid being distilled is obviated.

Five grammes of the butter-fat are saponified, the soap dissolved in 150 c.c. of boiling water, 50 c.c. of 5 per cent. sulphuric acid introduced, and 100 c.c. of the liquid distilled in thirty to thirty-five minutes. The distillation is repeated after the addition of 100 c.c. of boiling water. The two distillates are filtered, and 50 c.c. of the filtrates titrated. The filters are washed with 50 c.c. of alcohol, and the washing mixed with the other 50 c.c. of the filtrates and again titrated.

In this way the following results, in c.c. of $\frac{N}{10}$ sodium hydroxide, were obtained :

	Soluble Volatile Acids.			Insoluble Volatile Acids.		
	1st Distillation.	2nd Distillation.	Total.	1st Distillation.	2nd Distillation.	Total.
Cocoanut oil	7·1	4·3	11·4	7·85	7·55	15·4
Butter, I.	22·4	5·4	27·8	0·6	0·3	0·9
,, II.	21·2	5·0	26·2	0·6	0·3	0·9
,, III.	23·8	5·2	29·0	0·4	0·4	0·8
Margarine	0·2	0·2	0·4	0·5	0·05	0·55
Mixture of 75 per cent. of butter (I.) and cocoanut oil, 25	18·4	5·8	24·2	2·8	2·2	5·0

The theoretical values for the mixture were: Soluble volatile acids, 23·7; and insoluble volatile acids, 4·55.

C. A. M.

The Reichert-Meissl Value of Dutch Butter. **L. T. Reicher.** (*Zeit. angew. Chem.*, 1901, 125-129.)—The abnormal composition of Dutch butter in the autumn was attributed by van Rijn to the cows being left too long in the fields, and thus exposed to the influence of bad weather and changes of temperature, and he considered that abnormalities might be prevented by an earlier stalling.

This question has been further investigated in the State Laboratory at Amsterdam from 1894 to 1901. The butter was prepared each week from the mixed milk of eight cows during 1894-98, and of two herds of twenty and twenty-five respectively during 1898-1901.

During the autumn months Reichert-Meissl values lower than at other times of the year were obtained, whilst the rise observed by van Rijn after stalling also occurred in the majority of cases. C. A. M.

The Detection of Archil in Wine. R. Truchon. (*Ann. de Chim. Anal.*, 1900, v., 444, 445.)—Fifty c.c. of the wine are acidified with 1 c.c. of 10 per cent. sulphuric acid and boiled for five minutes with a little wool, which is then washed and immersed in ammoniacal water. In the case of natural wines the wool assumes a green tint, whilst if the wine contained archil it becomes violet, the intensity of the shade depending on the quantity of colouring matter. C. A. M.

Detection of Foreign Colouring Matter in Spirits. Second Method. C. A. Crampton and F. D. Simons. (*Journ. Amer. Chem. Soc.*, xxii., 810.)—The authors prefer the following method to their first one (*Journ. Amer. Chem. Soc.*, xxi., 355),* which depends on the absorption of caramel by Fuller's earth. The present method is based on the insolubility in ether of the foreign colouring matter of caramel and prune-juice, the natural colouring matter of oak-wood being readily soluble in ether. Fifty c.c. of the sample are evaporated on the water-bath nearly to dryness; the residue is washed into a 50 c.c. graduated flask, and 25 c.c. of absolute alcohol are added; the solution is then made up to the mark with water. Twenty-five c.c. of this solution are then transferred to a separating funnel, and shaken up with 50 c.c. ether at intervals for half an hour. A special separating apparatus is used, having two bulbs connected by a long stem, on which is the graduating mark of the lower bulb, which holds exactly 25 c.c.; the upper bulb has a capacity of about 100 c.c. When the extraction is finished, the lower layer is again made up to 25 c.c. with water. The two layers are again mixed; the lower one is drawn off, and its colour compared in a tintometer with that of the other 25 c.c. of the original solution. The amount of colour extracted by the ether is then calculated in percentages. To obtain accurate results, the conditions given should be carefully adhered to.

In a series of thirty-four naturally-coloured spirits subjected to the above treatment, on an average 41·7 per cent. of the colour was removed by the ether (max. 51·1, min. 36·4 per cent.). In a series of seventeen artificially-coloured spirits, on an average 14·8 per cent. was removed (max. 23·2, min. 0·0 per cent.). A. G. L.

The Assay of Coca. W. R. Lamar. (*Amer. Journ. Pharm.*, 1901, lxxiii., 125-131.)—In the author's opinion, the main cause of the many discordant results obtained in the analyses of this drug is the want of appreciation of the instability of the various alkaloids.

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* Analyst, xxiv., 207.

Cocaine, cinnamyl-cocaine and isatropyl-cocaine are all methyl esters of ecgonine, and the methyl group attached to the carboxyl is readily split off, forming, in the case of cocaine, benzoyl-ecgonine, which still possesses alkaloidal properties, but is practically insoluble in the ordinary solvents used to extract the alkaloids from their alkaline solution.

To obviate this, it is essential to avoid a large excess of alkali, and to have this of the lowest possible strength capable of completely liberating the bases.

The author has found petroleum a suitable solvent for extracting the alkaloids, since it removes the smallest possible amount of other matters. The following modification of E. Squibb's process is recommended:

Twenty-five grammes of the powdered leaf are well mixed with 25 c.c. of a 2 per cent. solution of ammonia, and left in a covered vessel for thirty minutes with an occasional stir. Then, if the odour of ammonia is still perceptible, 75 c.c. of petroleum are introduced little by little, with continual stirring, and the whole macerated for an hour or more, being stirred every ten to fifteen minutes.

The contents of the jar are next transferred to a percolator, the last portions of leaves being washed out with petroleum, fresh supplies of which are added when necessary, and about 450 c.c. of percolate collected. The percolate is shaken in a separating funnel with 25 c.c. of $\frac{N}{10}$ hydrochloric acid for ten minutes, the acid liquid withdrawn, and the shaking repeated twice more with the same quantities of acid. The united acid extracts are shaken with 20 c.c. of ether, and subsequently with a fresh portion of 15 c.c. of ether, so as to eliminate the last traces of petroleum and colouring matter.

The acid solution is then transferred to a third separating funnel. The ether in the first funnel is shaken twice with 5 c.c. of water, and the aqueous layers added to the ether in the second funnel, whence, after shaking and separating, they are drawn off into the third funnel, containing the main solution.

Sufficient ammonia solution (10 per cent., diluted with four times its volume of water) to render the liquid alkaline is now introduced, about 8 or 9 c.c. being required. The alkaloids are then extracted with successive portions of 40, 30 and 30 c.c. of ether, the united extracts evaporated in a weighed beaker at 30° to 35° C., and the residue dried at 60° C. until constant in weight. The alkaloids thus obtained are crystalline and nearly colourless. As a check upon the weight, they may be titrated by using an excess of $\frac{N}{20}$ sulphuric acid, and titrating the excess of acid with $\frac{N}{20}$ potassium hydroxide solution using cochineal tincture as indicator. The mean factor for the alkaloids, as found in numerous experiments, was 0·01514 gramme for each c.c. of $\frac{N}{20}$ sulphuric acid.

A. Dohme (*Amer. Pharm. Assoc.*, 1895, 268) claimed that the Keller method was the best method of assaying coca. The author, however, finds that although the latter method gives higher gravimetric results, it gives much lower ones by titration, and the alkaloids are dark brown in colour and crystallize with difficulty. This is attributed to the mixture of chloroform and ether extracting substances which the petroleum does not. From numerous assays the author concludes that coca-leaves of good quality should yield, by the process described above, about 0·7 per cent. of total alkaloids.

C. A. M.

Commercial Assafœtida. M. I. Wilbert. (*Amer. Jour. Pharm.*, 1901, lxxiii., 131-135.)—The author has obtained the subjoined results in the examination of different samples of the drug as sold in the American market:

Description.	Soluble in Alcohol per cent.	Insoluble in Alcohol per cent.	Ash per cent.
1. Loose tears	70·1	29·9	7·2
2. Lump	44·3	55·7	34·2
3. Choice gum	41·4	58·6	35·8
4. Mass tears	36·4	63·6	45·1
5. Lump	31·2	68·8	51·9
6. ,, ...	30·2	69·8	50·6
7. Powdered	28·5	71·5	46·6
8. ,, ...	19·8	80·2	60·6
9. Soft mass	18·3	81·7	62·1
10. Old gum	40·5	59·5	45·9

The author considers that the American drug is grossly adulterated, and seldom complies with the Pharmacopœial requirements. C. A. M.

ORGANIC ANALYSIS.

A Generally Applicable Volumetric Method for the Determination of Aldehydes. Maximilian Ripper. (*Monatshefte f. Chem.*, xxi., 1079.)—The method is based on the combination of alkaline bisulphites with aldehydes. Twenty-five c.c. of the solution to be examined, which should not contain more than ½ per cent. of the aldehyde, are run into 50 c.c. of a solution of potassium bisulphite containing 12 grammes $KHSO_3$ per litre, placed in a 150 c.c. flask, which is then securely corked, and allowed to stand for a quarter of an hour. During this time another 50 c.c. of the potassium bisulphite solution are titrated with decinormal iodine solution. The excess of bisulphite added to the aldehyde solution is then determined with the same iodine solution, and from the difference the amount of aldehyde present is calculated.

Solutions of potassium bisulphite stronger than the above should not be employed, as the larger quantities of hydriodic acid formed would exert a reducing action on the sulphuric acid formed. The use of alcohol to dissolve the aldehyde should also be avoided as far as possible, as even relatively small quantities of alcohol (upwards of 5 per cent.) prevent the iodide of starch reaction. With the very dilute solutions of aldehyde used, however, a very small addition of alcohol will be sufficient in most cases.

The method was tried with formaldehyde, acetaldehyde, benzaldehyde, and vanillin. For the last three, the author prepared the pure aldehydes from commercial specimens, and then made up solutions of known strength. In the case of formaldehyde, he used the hexa-methylene-tetramine method to check his results. In all cases satisfactory results were obtained. A. G. L.

The Valuation of Commercial Solutions of Lactic Acid. **F. Jean.** (*Ann. de Chim. Anal.*, 1900, v., 285, 286.)—From 5 to 10 c.c. of the solution are used for the determination of the total acidity in terms of sulphuric acid.

A similar quantity of the sample is evaporated on the water-bath, the residue mixed with 10 c.c. of water, and again evaporated, this process being once more repeated, so as to expel all volatile acid. The fixed acidity is then determined in the residue.

From 5 to 10 c.c. of the original solution are evaporated to dryness, the residue taken up with water, and the liquid brought to boiling-point, and neutralized with precipitated barium carbonate added little by little. The filtrate is evaporated in a platinum basin, and the residue ignited at a moderate temperature, so as to convert the barium lactate into carbonate. It is then treated with boiling water, and the insoluble portion collected on a filter, and washed with boiling water until the washings cease to be alkaline. A definite quantity of standard hydrochloric acid is then run on to the filter, so as to dissolve the barium carbonate, and the filtrate, after being boiled, titrated with standard alkali, the result giving the amount of hydrochloric acid neutralized by the barium carbonate derived from the barium lactate.

Ten c.c. of $\frac{N}{10}$ hydrochloric acid correspond to 0·09 gramme of lactic acid.

C. A. M.

Separation of Oxalic Acid from Tartaric Acid. **M. Palladini.** (*Gazz. Chim. Ital.*, 1900, xxxii., [2], 446; through *Chem. Zeit. Rep.*, 1901, 33.)—When attempts are made to separate these acids by precipitating with calcium chloride in presence of acetic acid, the calcium oxalate carries some tartrate down with it; and the latter cannot be entirely extracted, even by boiling with a large excess of acetic acid. The process is therefore not adapted for quantitative purposes. F. H. L.

Examination of Wax. (1) **J. Werder.** (*Chem. Zeit.*, 1900, xxiv., 967.) (2) **K. Dieterich.** (ANALYST, 1897, xxii., 242.)—Werder finds that the Hübl prescription —*i.e.*, heating 5 grammes of wax for forty-five minutes on the water-bath with alcoholic alkali—is not sufficient to insure thorough attack of the beeswax; and by operating in this manner, he shows that the saponification value, the ester number, and the "ratio" are not given correctly. He recommends boiling the mixture for one hour in a flask with inverted condenser over a naked flame and wire-gauze. By taking 5 grammes of wax, 25 c.c. of seminormal potash, and 20 c.c. of 95 per cent. alcohol, the process can be combined with Weinwurm's qualitative test for paraffin and ceresin (ANALYST, 1897, xxii., 242), for the reaction appears with less than 5 per cent. of either adulterant when the alcoholic liquid is diluted with hot water, and there is no necessity to remove the spirit and add glycerin as proposed by Weinwurm.

By weighing the amount of matter soluble in ether after saponification of bleached or unbleached wax, the proportion of paraffin or ceresin may be determined, for after examining some twenty samples of genuine wax, the author finds that between 48·55 and 53·01 per cent. of unsaponified matter always remains. Two grammes of the sample are boiled as above with 5 c.c. of normal potash and 15 c.c. of alcohol, the solution is concentrated in a basin to about 15 c.c., the residue mixed with sand, dried, and extracted for three hours with pure dry ether, drying the fat for one hour at 100° C. Occasionally the wax may contain so little unsaponifiable matter that an addition of 5 per cent. of ceresin or paraffin might not be shown by this quantitative process; but its presence would be indicated by the qualitative test.

Dieterich agrees that the Hübl directions are unsatisfactory. In the interest of rapidity, he prefers to use only 3 grammes of wax, and to boil for half an hour. Both authors regret that some standard methods of analysing wax have not been decided upon, regarding uniformity as important. F. H. L.

Notes on the Estimation of Pentosans. G. Kröber. (*J. Landwirthsch.*, 1901, xlviii., 357; through *Chem. Zeit. Rep.*, 1901, 40.)—The author has submitted the hydrochloric acid and phloroglucinol method of estimating pentosans to a further examination. He finds that the presence of di-resorcinol in the phloroglucinol is absolutely without influence on the weight of phloroglucide obtained. The latter should be dried for four hours; and as it is very hygroscopic, it must be weighed in a closed tube. Repeated or slow desiccation does not alter its weight, provided moisture be excluded. Exposed to the air, the phloroglucide gains in weight, and retains that gain even when dried once more. This, however, is due to the fact that it oxidizes somewhat during the second drying, and therefore it must certainly oxidize a little during the first desiccation. The solubility of the phloroglucide in 550 c.c. of liquid (400 c.c. of dilute hydrochloric acid and 150 c.c. of water) is 5·1 to 5·2 milligrammes. Ninety-six parts of furfural give 184·9 parts of phloroglucide; and this is practically the theoretical yield, the molecular weight of the substance being 186 ($C_{11}H_6O_3$). Only the one condensation product is formed between phloroglucinol and furfural. The original paper includes tables for converting the yield of phloroglucide into furfural, arabinose, or xylose, with the corresponding quantities of pentosans, etc. F. H. L.

Volumetric Estimation of Tannin. L. Specht and F. Lorenz. (*Chem. Zeit.*, 1901, xxv., 5.)—This article describes certain modifications and further investigations into the method already recorded (ANALYST, 1900, xxv., 163). To simplify the process, and to eliminate the disturbing influence of the oxygen dissolved in the water, the operation is conducted as follows: 0·45 gramme of tannin, 0·54 gramme of tartar emetic, and 0·90 gramme of safranine, are precipitated in 1,500 c.c. of water; and then 35 c.c. of sodium bisulphite and 33 c.c. of sodium bicarbonate, each in 10 per cent. solutions, are added. The titration, etc., remains as before prescribed.

Thus carried out, the method generally gives high results which vary in inaccuracy according to mass conditions. The present paper contains a mathematical investigation of the sources of error, and formulæ for their separate elimination. Briefly, the combined errors may be removed by the use of the annexed table:

True Tannin, per Cent.	Tannin by Analysis, per Cent.	Correction per 0·5 per Cent.
100	100·0000	
		0·37823
95	96·2177	
		0·39294
90	92·2883	
		0·40682
85	88·2201	
		0·42016
80	84·0185	
		0·43240
75	79·6945	
		0·44395
70	75·2550	
		0·45510
65	70·7040	
		0·46590
60	66·0450	

The table is employed thus: With a tannin of known purity (100 per cent.), titration shows that the "lake" contains (say) 0·68712 gramme of safranine. The same weight of tannin to be examined gives a lake containing (say) 0·54803 gramme of dye; whence its percentage purity is (0·68712 : 0·54803 : : 100 :) 79·757. Applying the correction in the table, this becomes

$$75+\frac{79\cdot757\times79\cdot6945}{0\cdot4324}\times0\cdot5=75\cdot072 \text{ per cent.}$$

The titration requires some experience to carry out successfully, for at first the operator is apt to use too much hydrosulphite. The liquid needs stirring as thoroughly as possible without causing the formation of bubbles. With low-grade specimens it is advisable to increase the weight taken until the amount of true tannin present roughly corresponds to that in the standard sample. The method has only been studied from the dyer's and printer's point of view, and on such materials only as are commonly employed by them.

F. H. L.

The Detection of Antithermic Substances in Urine. **A. Petermann.** (*Ann. de Chim. Anal.*, 1901, vi., 4-6.)

Salicylic Acid given in the Form of Sodium Salicylate, or Salol.—These are taken as typical of the compounds of salicylic acid which are decomposed in the animal organism with the liberation of salicylic acid. The urine should be rendered slightly acid, but too much acid interferes with the ferric chloride test, and may produce by itself a reddish-orange colour. One drop of a 10 per cent. solution of ferric chloride is added to 25 c.c. of the urine.

Antipyrine.—When the urine, tested as described above, gives, instead of a violet coloration, a blood-red colour, which disappears on the addition of an excess of acid or alkali, the presence of antipyrine is probable. It is confirmed by extracting the urine with chloroform or benzene.

Colouring Matters of the Urine.—Not infrequently urine gives a coloration with ferric chloride which might be confused with the reactions for salicylic acid and antipyrine. It differs from these, however, in not being destroyed by hydrochloric acid, and in eventually yielding under the above conditions a precipitate of the same colour. C. A. M.

Quantitative Reactions distinguishing between Petroleum and Brown Coal-Tar Pitch, and the Pitchy Residue from the Distillation of Fats. **D. Holde.** (*Berichte*, 1900, xxxiii., 3171-3175.)—Although the hard pitchy residues left on distilling fats contain only a trifling proportion of free fatty acids and esters, it is possible to concentrate these by extracting the substance with a mixture of alcohol and ether, and then to obtain solutions giving considerable higher acid and ester values than the corresponding extracts of petroleum and brown coal-tar pitches.

About 20 grammes of the pitch are dissolved, as far as possible, in 80 c.c. of ether eventually by boiling under a reflux condenser. The bulk of the neutral black constituents are precipitated by the addition of absolute or 96 per cent. alcohol, the liquid filtered after some time, and the filter washed with a mixture of alcohol and ether.

The filtrate is evaporated, and the residue dried and weighed. It is then re-dissolved in ether containing a little alcohol, and the acid value determined by titration with $\frac{N}{10}$ alkali.

The ester value is determined by evaporating the solution and boiling the residue with 25 c.c. of $\frac{N}{10}$ alcoholic alkali and 25 c.c. of benzene, and titrating the excess of alkali with hydrochloric acid. A 2 per cent. alcoholic solution of alkali blue 6*b* is used as indicator, and the end-point found by shaking the liquid in a flask, and noting the colour of the liquid running down the sides.

As a further test the pitch is distilled either alone or with superheated steam, in which process the fatty acids are concentrated in the first fraction (one-fourth) of the distillate, which consequently gives a higher acid value than that of the corresponding fraction from petroleum or tar-pitch.

The following results were thus obtained:

DRY DISTILLATION. ACID VALUES.

	Fraction I. About one-fourth.	Fraction II. About one-half.	Fraction III. About one-fourth.
Hard wool-fat pitch ...	5·2	1·1	0·08
Mixture of hard fat pitches	5·3	0·95	0·6
Hard petroleum pitch ...	0·4	0·4	0·3
Brown coal-tar pitch ...	0·1	0·2	0·4
Brown coal-tar pitch II. ...	0·2	0·6	0·6

DISTILLATION WITH SUPERHEATED STEAM. ACID VALUES.

	Fraction I.	Fraction II.	Fraction III.
Mixture of hard fat pitches	14·6	13·7	13·4
Soft wool-fat pitch ...	34·8	37·8	7·0

The author remarks that all the fat-pitches examined by him contained traces of copper derived from the copper distillation vessels, whilst the petroleum pitches (which are distilled in iron vessels) were all free from copper. C. A. M.

INORGANIC ANALYSIS.

The Separation of Cerium Earths in Monazite Sand. R. J. Meyer and E. Marckwald. (*Berichte*, 1900, xxxiii., 3003-3013.)—From the results of their experiments with different methods the authors recommend the following process as the most suitable. The original material used by them was purchased as "cerium oxalicum oxydul pur." containing about 25 per cent. of water.

Conversion of the Oxalates into Soluble Nitrates.—When the oxalates are ignited in air, a mixture of oxides is obtained, the solubility of which depends upon the proportion of cerium, and which can only be dissolved with great difficulty when certain other substances are present. This can be obviated in the following way: On boiling the mixed oxalates with potassium hydroxide solution in the presence of hydrogen peroxide, the oxalic acid is completely eliminated, and a yellow crystalline precipitate is obtained, consisting of a mixture of ceric hydroxide with higher oxides (? CeO_3). This is drawn off by suction, washed, dried at 120° C., and dissolved in concentrated nitric acid, yielding a dark-red solution. The drying at 120° C. must be continued for some time in order to destroy the cerium peroxides, since these would subsequently act like hydrogen peroxide and reduce the ceric solution.

If it is not desired to obtain a solution containing the cerium in the tetravalent condition, but to oxidize it subsequently as in the method of Witt and Theel, the oxalic acid is best removed by boiling the mixed oxalates with concentrated nitric acid. For this purpose the oxalates are gradually introduced into twice their weight of nitric acid (specific gravity 1·4), which is boiled, with the addition of a few drops of nitric acid from time to time, until the evolution of gas has ceased. Intermediate products consisting of compounds of oxalates and nitrates are first formed, and if the boiling has not been continued long enough, separate out on cooling.

To the brown solution of nitrates thus obtained ammonium nitrate is added in the proportion of 80 grammes to 100 grammes of the original mixture, and on evaporating the liquid to a third of its volume, the double nitrates crystallize out. They are much less hygroscopic and less soluble than the simple nitrates. The crystals are dissolved in water, and the solution used for the separation of ceria.

Separation and Purification of Ceria.—In the authors' opinion the method of O. N. Witt and W. Theel (*Berichte*, xxxiii., 1315) is likely to completely supersede the older methods. They have not succeeded, however, in completely freeing the precipitate (which contains the entire cerium in the form of basic sulphate) from other earths by washing. The washed precipitate, which consists of basic ceric sulphate and calcium sulphate, with traces of didymium and lanthanum salts, is dried at 120° C., powdered and dissolved in ten times its weight of hot concentrated nitric acid (1·4), which has previously been freed from nitrous oxides by boiling. Ammonium nitrate dissolved in a little water is added to the dark-red solution in the proportion of about one and a half times the quantity of the dissolved precipitate, and the double nitrate $[(NH_4)_2Ce(NO_3)_6]$ soon crystallizes out, a further portion being obtained on evaporating the mother-liquid. The double nitrate is purified by recrystallizing once or twice from concentrated nitric acid (1:4), drained by suction on a platinum cone, and dried over sulphuric acid and potassium hydroxide.

Separation of the Yttrium Earths.—As monazite sand only contains small quantities of these, it is sufficient to effect the separation by the addition of finely pulverized potassium sulphate to the *boiling* solution (Muthmann and Rölig, *Berichte*, xxxi., 1722). The filtrate from the cerium precipitation is heated to boiling, and a strong current of steam introduced, whilst the potassium sulphate is introduced little by little, until a spectroscopic test of the solution shows that didymium has practically disappeared. About 125 grammes of potassium sulphate are required for 400 grammes of the original crude oxalate. The clear supernatant liquid is decanted whilst hot, and the precipitate washed several times with a cold aqueous solution of potassium sulphate, dried, and weighed.

Conversion of the Double Sulphates of Didymium and Lanthanum into Nitrates.—The method which the authors have found to be the most simple is to boil the sulphates for a short time with five times their weight of concentrated nitric acid. The entire mass is rapidly poured into boiling water (1500 c.c. to 100 grammes of sulphates), and the clear solution thus obtained treated with ammonium oxalate (75 grammes to 100 grammes of sulphates), and then neutralized with concentrated ammonium hydroxide. The precipitated oxalates are withdrawn by suction, introduced into one and a half times their quantity of concentrated nitric acid, and converted into nitrates, as described above. The acid solution of nitrates is evaporated in the water-bath until nitric acid ceases to be evolved. The residual syrup is dissolved in hot water and diluted to a litre.

Separation of Didymium and Lanthanum.—In the authors' opinion the method of Muthmann and Rölig (*loc. cit.*) is the most simple and exact for this purpose. It consists in fractionally precipitating the boiling, nearly neutral, solution of didymium and lanthanum with magnesia, in which process the didymium earths, being the less basic, are precipitated first. This precipitation should be twice repeated.

The sample of crude oxalate thus analysed by the authors had the following composition: Water, 25·0; cerium oxalate (anhydrous), 32·5; didymium oxalate (anhydrous), 21·5; lanthanum oxalate (anhydrous), 14·0; and yttrium earths and loss, 7·0 per cent. C. A. M.

Irregular Distribution of Sulphur in Pig Iron. Randolph Bolling. (*Journ. Amer. Chem. Soc.*, xxii., 798.)—To study the irregular distribution of sulphur in pig-iron, a rod 12 × 14 inches, and weighing about 10 pounds, was cast (vertically) in sand. Eight holes were drilled at intervals of 1⅝ inches, starting from the bottom, and sulphur determined by the cadmium chloride method. The results expressed in percentages were: 0·023, 0·03, 0·032, 0·036, 0·036, 0·036, 0·036, 0·036, showing a difference of 0·013 per cent. between the upper and lower surface. To obtain a true sample, it would consequently be necessary to drill a hole right through the rod, and mix the drillings well. In another case, drillings from the top surface showed 0·075 per cent. sulphur, whilst drillings from a hole through the sample gave 0·045 per cent. A. G. L.

A Method for the Rapid Determination of Carbon in Steel. Robert Job and Charles T. Davies. (*Journ. Amer. Chem. Soc.*, xxii., 791.)—The authors have simplified G. W. Sargent's method (ANALYST, xxv., 302). The combustions are carried out in a porcelain tube 20 inches long, which is heated by three burners having spreaders on their tops, and contains a roll, 4 inches long, of oxidized copper gauze, kept in position by placing pieces of clay-pipe stems between it and the end of the tube. The products of combustion pass first through a U-tube, one limb of which is filled with anhydrous cupric sulphate, the second limb containing anhydrous cuprous chloride. This tube may be regenerated by heating whilst applying a gentle suction. The gases then pass through a bubble-tube containing 5 to 10 c.c. of a solution of silver sulphate in sulphuric acid (specific gravity 1·40), which is refilled if any precipitate forms; next through a calcium chloride tube, and then into the weighed potash bulbs. To prevent the boats from cracking the tube, a cylinder of platinum foil is placed in the tube.

Oxygen free from hydrocarbons is readily obtainable, and need only be passed through potash bulbs to purify it. Should it be necessary, however, to pre-heat it, this may be done in a copper tube placed in the same furnace above the porcelain tube. During the first part of this combustion oxygen is passed at the rate of four bubbles per second for seven minutes, and then air at the rate of six or seven bubbles per second for twelve and a half minutes. Three weighed bulbs are used for a set of determinations. The bulbs are allowed to stand for forty minutes in the balance-case before weighing.

A. G. L.

Kilgore's Modification of the Volumetric Method of estimating Phosphoric Acid in Fertilizers. C. B. Williams. (*Journ. Amer. Chem. Soc.*, xxiii., 8.)—By this method the author was able to deal with thirty samples daily with great facility. For totals, 2 grammes of the fertilizer are boiled down with 30 c.c. concentrated nitric acid and 10 c.c. hydrochloric acid in a 200 c.c. flask to a concentration of 8 to 10 c.c., unless the sample contains much iron and alumina, in which case it is first boiled for half an hour with 30 c.c. hydrochloric acid, 30 c.c. nitric acid are then added, and the boiling continued until the excess of hydrochloric acid is removed. After cooling, the solution is made up to the mark and filtered, or allowed to clear by standing. Twenty c.c. are then measured into a 500 c.c. Erlenmeyer flask, and 10 to 12 grammes ammonium nitrate and 50 c.c. water added; the excess of acid is neutralized with ammonia, and, when cold, 300 c.c. of clear molybdic solution are added, and the flask is placed in a shaking machine, maintained at forty-five to fifty-five revolutions per minute, for thirty minutes. The solution is then filtered through a perforated porcelain disc covered with asbestos, and placed in a carbon filter, and flask and precipitate thoroughly washed. The outside of the filter is then washed free from acid, the filter inverted over the flask in which the precipitation was effected, and the disc and asbestos are then pushed into the flask by means of a copper-wire rigidly connected to the disc, and reaching through the stem of the funnel. The contents of the flask are then titrated, using solutions of potassium hydroxide and nitric acid, 1 c.c. of which corresponds to 0·5 milligramme of phosphoric acid.

For insolubles, 40 c.c. of solution are taken; the process is carried out as above, except that little or no water is added before precipitation.

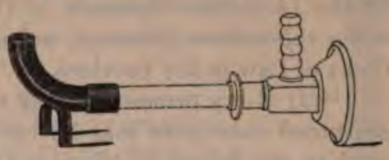
A large number of determinations were made, in which the above method was compared with the official gravimetric method. In all instances the results were extremely satisfactory.

A. G. L.

APPARATUS.

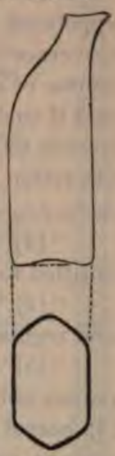
A Burner for Use in Confined Situations. W. Burstyn. (*Oesterr. Chem. Zeit.*, 1900, iii., 518.)—As shown by the cut, this device consists of a brass "bend," with socket attached, which can be slipped over an ordinary Bunsen when laid on its side. With its aid vessels can be heated that are more conveniently placed close to the bench, while it also permits the flames of several burners to be brought quite close together.

F. H. L.



A New Shape for Reagent Bottles. B. Setlik and J. Fric. (*Chem. Zeit.*, 1900, xxiv., 1138.)—The construction of these bottles is shown in elevation and cross-section by the annexed illustration. It is easier to pour from them when they are quite full; to obtain single drops of liquid from them; to empty them to the last drop; and to charge burettes and similar narrow vessels from them, than with the ordinary cylindrical bottles having concentric necks.

F. H. L.



BUREAU OF MINES AMENDMENT ACT OF BRITISH COLUMBIA.

This Act, which forms a new and important departure in legislation, is given *in extenso*. So far as British Columbia is concerned, analytical chemists are placed on a par with the members of the legal and medical professions, insomuch that no analyst henceforth will be allowed to practise in that colony unless he has satisfied the examiners appointed under the Act as to his skill and competence. The principle involved is one that might be adopted in other countries with advantage.

CHAPTER XI.

AN ACT TO AMEND THE "BUREAU OF MINES ACT."

27th February, 1899.

Her Majesty, by and with the advice and consent of the Legislative Assembly of the Province of British Columbia, enacts as follows:

1. This Act may be cited as the "Bureau of Mines Act Amendment Act, 1899."

2. Section 12 of Chapter 36 of the Revised Statutes, 1897, is hereby repealed, and the following substituted therefor:

"12. (1) The Minister shall, twice or oftener in each year, institute an examination for efficiency in the practice of assaying and other kindred subjects as may be deemed advisable; the examination shall be conducted by examiners to be appointed by the Minister; each candidate shall pay, before the examination, such fee as may be determined by the Lieutenant-Governor in Council, who shall grant a certificate of efficiency to each successful candidate at such examination, on the recommendation of the duly appointed examiners, and the payment of a further fee to be determined by the Lieutenant-Governor in Council, such certificate, and the certificate issued under sub-section (2) hereof, to be taken as *primâ facie* evidence of efficiency in any Court of Law in the Province.

"(2) Every person applying for a certificate of efficiency, on satisfying the duly appointed examiners that he has passed a course of practical analytical or assay work in any School of Mines or College in Canada, Great Britain, or Ireland, shall be entitled to receive a certificate of efficiency on the recommendation of the duly appointed examiners, and on payment of a fee to be determined by the Lieutenant-Governor in Council; but it shall rest with the examiners to decide whether such course or examination is equivalent to the examination prescribed by the examiners, and if such examination or course is not, in the opinion of the examiners, sufficiently similar to the examination required by the examiners, the examiners may, in their discretion, require such candidate to pass such further examination as they may deem necessary.

"(3) The examiners appointed by the Lieutenant-Governor in Council shall be entitled to receive a certificate of efficiency.

"(4) The Lieutenant-Governor in Council may from time to time prescribe rules and regulations for the carrying out of such examinations.

"(5) After the expiration of two years from the first day of March, A.D. 1899, no person not holding a certificate of efficiency as prescribed in sub-sections (1), (2), and (3) hereof shall, by public advertisement or otherwise, solicit employment as an assayer, or to perform assays, nor shall any such person ask or receive payment for performing any assay, nor shall any such person give a written statement of the result of any assay performed by him: Provided that nothing in this sub-section shall apply to persons who already hold a certificate of efficiency under the examination prescribed by section 12 of the 'Bureau of Mines Act.'

"(6) Any persons violating the provisions of this section shall be liable to a fine of not less than twenty-five dollars and not exceeding one hundred dollars for each offence."

APPOINTMENT.

Mr. J. CUTHBERT WELCH, of Trail, has been appointed one of the three examiners under the Bureau of Mines Amendment Act of British Columbia of 1899.

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THE ANALYST.

JUNE, 1901.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE monthly meeting of the Society was held on Wednesday evening, May 1, in the Chemical Society's Rooms, Burlington House. The President (Dr. J. Augustus Voelcker, M.A., B.Sc.) occupied the chair.

The minutes of the previous meeting were read and confirmed.

Mr. John Webster was elected a member of the Society.

A certificate of proposal for election to membership in favour of Mr. Frederick Malcolm Wharton, F.I.C., 58, Belsize Road, South Hampstead, assistant to Mr. William Chattaway, and late assayer, Imperial Mint, Nanking, China, was read for the first time.

The following papers were read: "Alkaline Waters from the Chalk," by W. W. Fisher, M.A.; "Citron Oil," by Herbert E. Burgess; and a "Note on the Reduction of Iron Salts," by Gilbert T. Morgan, D.Sc.

DETERMINATION OF DISSOLVED OXYGEN IN WATERS IN PRESENCE OF NITRITES AND OF ORGANIC MATTER.

BY DR. S. RIDEAL AND C. G. STEWART.

(*Read at the Meeting, March* 6, 1901.)

ACTUAL boiling out of the dissolved gases from water with gasometric estimation can in most cases only be accomplished in the laboratory. Bunsen's method, described in Fresenius, is fairly simple, but not accurate; his absorption coefficients have been re-estimated and corrected by Pettersson and Sonden (*Berichte*, xxii., 1439), by Winkler (*ibid.*, 1764), and by Roscoe and Lunt (*Trans. Chem. Soc.*, 1889, 552) Various forms of special apparatus have been devised by Pettersson and Sonden (*loc. cit.*), Sidney Harvey (ANALYST, 1894, 121), Adeney (*Trans. R. Dubl. Soc.*, Sept., 1895), Dibdin (*J. S. C. I.*, 1900, 499), and others, but none of them are suitable for use in the field.

Many titration processes have been introduced for rapidly estimating the dissolved oxygen only, but with heavy and fragile apparatus, while some, *e.g.*, Schutzenberger's and Thresh's, require the use of a current of coal gas or hydrogen. Kisch, in an examination of different methods (*Zeits. f. angew. Chem.*, 1891, 105), strongly prefers Winkler's, which employs manganous chloride and soda (*Ber.*, xxi., 2843; xxii., 1764). Müller introduced some changes in the preceding, using crystals of manganous sulphate and allowing to stand with the alkali for half an hour. He mentions the interference of nitrites, and corrects for it by titration of a separate sample with HCl, KI and thiosulphate (*Chem. Zeit.*, xiii., 1188; *J. S. C. I.*, 1889, 923). Romijn keeps the manganous hydroxide in solution by adding Rochelle salt before the soda (*Rec. Trav. Chim.*, 1896, xv., 76). The use of ferrous hydroxide as an oxygen absorbent was antecedent to manganous, and was due to Mohr; it has led to a number of modifications, chiefly in the apparatus, but in every case the instability of ferrous salts renders them less suitable than manganous solutions for field work, and involves extra titrations of the standard. The sample of water, and at the same time an equal quantity of water freed from dissolved oxygen, are treated with ferrous sulphate and soda; after agitation, air being excluded, the precipitated hydroxides are dissolved by sulphuric acid, and the amount of iron oxidized is estimated by titrating both with standard permanganate.

Tiemann and Preusse (*Ber.*, xii., 1768), from a large number of trials, concluded that Mohr's method gave generally too low results, while König and others (*Ber.*, x., 2017; 1880, 154; *Zeit. Chim.*, xix., 259) found it trustworthy.

Lalieu (*Journ. Pharm. Anvers*, 1887, 570) employed ammonio-ferrous sulphate as more stable; the presence of ammonium salts also causes part of the ferrous hydroxide to remain dissolved, and renders the absorption more rapid. He used 200 c.c. of water in a narrow-necked flask with a perforated stopper.

Blarez (*Journ. Pharm.*, 1888, 5, xviii., 55) introduced the use of two superposed bulbs with a capillary tube between and a stopcock below, drawing in the reagents from above by running out mercury placed in the lower bulb; the apparatus was thus complicated and the weight increased.

Linossier (*Journ. Pharm.*, 1891, 5, xxiii., 103) avoided precipitation by the presence of sodio-potassic tartrate, and titrated the free oxygen with standard ferrous sulphate, with phenosafranin as indicator. The process, as described, has several faults, and is unsuited for the field.

Albert Levy, at the Montsouris Observatory, Paris, has in practice a modification of the Mohr-Lalieu method, employing a bulb tube of known capacity, "about 100 c.c.," with stopcocks above and below, and a short funnel above. The bulb being filled with the water, and the taps closed, 2 c.c. of potash solution are added through the funnel by carefully opening the cocks; then, after washing the funnel, 4 c.c. of ammonio-ferrous sulphate are similarly introduced. After agitation the precipitated oxides are dissolved by adding in the same way, after again washing the funnel, 2 c.c. of 50 per cent. sulphuric acid, the contents are transferred to a flask, and titrated with permanganate. The next step is the special feature. As the ferrous solution *in the acid state* only very slowly takes up oxygen, he repeats the whole operation, *omitting the alkali*; the difference between the c.c. of permanganate used is calculated into oxygen per litre.

Letts and Blake (*Brit. Assoc.*, 1900) fill an ordinary separating funnel with the water, withdraw a measured volume from below, and replace it by standard ferrous sulphate, and then ammonia. The funnel is inverted, and 50 per cent. sulphuric acid passed in by gravity through the tube. Permanganate is used for titration, or bichromate if the chlorides are very high, as in sewage or sea-water. But bichromate, equally with permanganate, is reduced by nitrites and by organic matter.

Thresh (*Trans. Chem. Soc.*, 1890, 185) adds a nitrite and sulphuric acid to the water in a current of coal gas; the iodine liberated is then proportional to the dissolved oxygen, and is measured by thiosulphate. Both the nitrite added and any originally present are separately titrated and allowed for. Organic matter interferes by absorption of iodine. Deventer and Jurgens substitute a layer of petroleum for the coal gas (*Chem. Centr.*, 1893, ii., 546).

Attempts were early made to utilize quantitatively in a direct manner the tints produced by free oxygen in various colourless solutions. This may be done in four ways:

I. Adding the reagents to a measured volume of water and comparing the tint with permanent standards. This, if reliable, would be a very rapid process. Professor Ramsay has recently introduced a very portable box, by which the colour produced by the dissolved oxygen acting on freshly-prepared ammonio-cuprous chloride is compared with standard blue tints preserved in sealed tubes. We received an early specimen of the apparatus, which in its present stage, after numerous trials, we have found to be subject to the following objections:

1. The standards from 1 c.c. to 6 c.c. should be extended to 7 and 8 c.c., as in cold weather many waters contain these quantities of oxygen per litre.

2. Readings are difficult nearer than ½ c.c., and in coloured waters cannot be defined.

3. It is very difficult to obtain a blank; the cuprous chloride in making absorbs oxygen, and the layer of kerosene over the water only partially excludes that gas.

4. Nitrites interfere greatly, as shown by the following experiments:

Note.—Two cylinders of equal diameter (3 centimetres) were filled to a depth of 6 centimetres with dilute colourless ammonio-cuprous chloride under 0·5 centimetre of petroleum, and left undisturbed. The liquid gradually became blue under the petroleum. The rate of penetration was as follows:

Hours	40 min.	1 hr. 40 min.	3	19	38	43	58
Depth of blue in centimetres:							
1st tube	0·63	0·84	1·0	1·9	3·8	4·6	5·9
2nd tube	nearly the same as first				2·9	3·6	3·8

At first the penetration is irregular, but afterwards it becomes approximately proportional to the time.

	C.c. Oxygen per Litre.		
	Theory, at 20° C.	Rideal-Winkler.	Ramsay.
Tap water alone	6·28	6·11	about 6
Ditto, with five parts nitrous N per 100,000 ...	6·28	6·15	much deeper blue
Water boiled and cooled under kerosene ...		0·78	1·5 to 2
Equal parts of boiled and tap water:			
Without nitrite	3·47	3·6	4·2 4·5 4·5
With nitrite	3·47		6·5 6·5 6·2

Ramsay's method therefore seems only applicable to fairly pure river or other waters. The presence of nitrates was not found to interfere.

II. Comparing the tint with standards made up at the same time as in Nesslerizing has not worked well in practice.

III. The colour produced by the oxygen is titrated back by means of a standard bleaching solution. Schutzenberger's indigo and hyposulphite process belongs to this category. Its difficulties and disadvantages are well known (Ramsay and Williams, *Journ. Chem. Soc.*, 1886, 751; Dupré, ANALYST, x., 156; Roscoe and Lunt, *loc. cit.*, 1889, 552; Adams, *ibid.*, 1892, 310).

IV. Adding the reagents to the sample, and to an equal volume of oxygen-free water, and admitting oxygen or air to the latter until the colour of the sample is imitated; the quantity of oxygen required gives the measure of that present in the specimen of water. This, which is really a titration with oxygen gas, presents many difficulties, and is liable to great errors. Mackay and Middleton (*Journ. Soc. Chem. Ind.*, 1898, 1127) add pyrogallic acid and potash to two equal samples of the water before and after boiling out the oxygen, then admit air gradually to the second till the tint of the first is obtained: as 250 c.c. of water are employed, the volume of residual nitrogen is taken as equal to the dissolved oxygen in c.c. per litre. Among the objections are the bulk and fragility of the apparatus, the time required, the use of coal gas, and the solubility of the nitrogen in the boiled water, a large quantity of which (265 c.c.) is required for each experiment. The analyses given in the paper are hardly satisfactory.

Some years ago we were led to select as the most portable, rapid, and generally reliable process a modification of Winkler's manganese method in preference to all the methods we have described. While it is comparatively easy to titrate the free oxygen in fairly pure samples, such as river and well waters, in sewage and effluents containing nitrites, occasionally sulphides, and much organic matter, great difficulties are introduced. After a large number of experiments, we have found that it is practicable to get rid of the interference by oxidizing with permanganate in acid solution out of contact with air. A further simplification was to discard tap funnels and indiarubber tubing,[*] and to conduct the experiment in ordinary stoppered bottles of known capacity (about 300 c.c.) when full, the volume of each being etched on the bottle and a coefficient calculated.[†]

With ordinary waters the proceeding is very simple. The bottle being filled quietly but quickly, 1 c.c. of manganous chloride solution (33 per cent.) is passed to the bottom from a long pipette, then 3 c.c. of a solution containing 33 per cent. soda and 10 per cent. KI. The stopper is inserted, without air bubbles, and the contents mixed by inversion and rotation. The liberated manganous hydroxide absorbs the free oxygen. On standing a few minutes the upper liquid becomes clear; the stopper is then removed for a second, and 3 c.c. of concentrated pure HCl are passed to the bottom by a pipette. Again closed and rotated, the manganese oxides dissolve and iodine is liberated in proportion to the oxygen. At this stage the bottle should be allowed to stand about five minutes in the dark, with occasional moving, till clear. The liquid is then transferred to a porcelain basin, and the iodine determined by thiosulphate and starch.

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[*] Roscoe and Lunt, in their examination of Schutzenberger's process with reduced indigo, found that indiarubber tubing allowed a considerable amount of oxygen to diffuse in from the air, also that, when an atmosphere of hydrogen or coal gas is employed, the oxygen rapidly diffuses into it, and then back again in an irregular manner.

[†] Zeitsche (*Chem. Centr.*, 1899, ii., 727) describes a similar simplification.

When nitrites and much organic matter are present, 50 c.c. of the original liquid, acidified with 1 c.c. of concentrated pure H_2SO_4, are first titrated with decinormal permanganate, till a faint pink colour persists after ten minutes. The number of c.c. so required is calculated to the volume of the bottle in which the free oxygen determination is to be done. This amount is then placed in the bottle, together with 1 c.c. of sulphuric acid (if more than 10 c.c. of decinormal permanganate are required, 2 c.c. of acid must be added), the bottle is filled with the liquid under examination, mixed by rotation, and allowed to stand five or ten minutes. We have found that it is advisable to add 0·1 c.c. of permanganate in excess of the calculated amount, so that the liquid should retain a slight pink colour. Any excess of permanganate affects the determination of free oxygen by liberating iodine, therefore has to be removed. If the solution remains pink, the bottle is briefly opened, ½ c.c. of a 2 per cent. solution of neutral potassium oxalate added with a pipette, the neck filled up with the water under examination, the stopper inserted, and the bottle rotated as before. The colour quickly disappears, and the solution is ready for the manganous chloride, etc., as in the ordinary determination. It was ascertained that the oxalate did not interfere, but to balance the sulphuric acid that has been added a rather larger proportion of soda is required. When working with sewages and effluents we use a 50 per cent. soda + 10 per cent. KI; after oxidizing with permanganate, 3 c.c. of this, and afterwards 3 c.c. of HCl, are added.

The reagents being used in the concentrated form with a comparatively large volume of water, the correction for the additions is small, and can usually be neglected. When, however, the oxygen is low, the reagents, being presumably saturated with oxygen under atmospheric conditions, will make the result too high. The correction then to be applied is:

$$x = \frac{1000\,a - \mathrm{R}n}{\mathrm{V} - n},$$

where x is the number of c.c. of oxygen per litre of the liquid, a the amount of oxygen in c.c. found by titration, V the volume of the bottle, and n that of the reagents, while R is the number of c.c. of oxygen contained in a litre of saturated water at the temperature of the experiment, which is preferably actually determined, or can be obtained from a table such as Roscoe and Lunt's (*C. S. J.*, 1889, 532).

The results obtained with good waters agree well together, and also with the figures recorded for boiling out *in vacuo*. Thus, in c.c. of O per litre a tap water in August, 1900, gave 5·93—corrected for reagents, 5·96; another observer found 5·97 uncorrected. Roscoe and Lunt's figure for saturated distilled water at the temperature 21° C., 6·16. Next day the tap water showed 6·23 and 6·15. In November determinations gave 7·24, 7·20 and 7·33. Roscoe's figure for 13·5° C. is 7·2.

In the presence of nitrite equal to 2 parts of nitrous nitrogen per 100,000, previous oxidation by permanganate gave, as c.c. of free O per litre:

	Water I.	Water II.
Original—nitrite absent	5·97	6·64
With addition of 2 parts nitrite per 100,000 ...	5·99 6·17	6·69

The following are other examples of the process:

	I.	II.	III.
Temperature	11·5° C.	14·0° C.	10·0° C.
Saturated water	7·54		7·74
Saturated water + 10 parts per 100,000 of N as nitrate		7·31	
Saturated water + 10 parts per 100,000 of N as nitrite, with previous permanganate treatment		7·26	7·62
Roscoe and Lunt's tables	7·52	7·40	7·77

In the presence of both organic matter and nitrite:—

A solution was made containing 10 per cent. of a very foul street washings, and 2 parts per 100,000 of nitrous N. This and an ordinary water were both saturated with oxygen by shaking. After the action on the polluted sample of permanganate, acid and oxalate as described, the dissolved oxygen was determined. The ordinary water gave 6·23, the polluted 6·29, 6·16, 6·19, and 6·39.

The quantity of nitrite was then doubled, =4 parts nitrous N per 100,000, about the maximum quantity we have found in effluents. The original gave 6·18 and 6·21 c.c. O per litre; the nitrited sample after the permanganating process gave 6·19.

The results should be expressed in actual c.c. of O found per litre, with the temperature of the water. The "percentage of saturation" calculated from Roscoe and Lunt's table should be appended.

Deviations from theoretical amounts may to a certain extent be produced by physical causes. With falling temperatures the water may not have had time to take up its complement of oxygen; on the other hand, with rising ones it may have an excess. Seyler and Gill (*Chem. News*, lxvii., 87) showed that the temperature might be raised from 1° to 9·5°, or from 13° to 26·5°, with little loss of oxygen, the water remaining supersaturated until shaken vigorously. This condition of supersaturation is not infrequent, especially if the water has been under pressure in pipes.

In a paper by Dr. Adeney* on "The Application of the Aeration Method of Analysis to the Study of River Waters," he points out (p. 361) that "the composition of the dissolved gases in still waters alters very slowly with alterations of temperature, so that samples of a good fresh river water may be, and probably always are, either oversaturated or undersaturated with oxygen for the temperatures of the water at the times of collection." This does not affect, however, the comparison of a series of samples collected at near the same time, if, as we advise, the amount of oxygen dissolved in local tap water be simultaneously ascertained.

Dr. Adeney in this paper gives a number of striking examples of the significance of free dissolved oxygen, as indicating the condition and progress of change in mixtures of sewage and river waters. He has made his determinations gasometrically, and includes the amount of nitrogen and carbonic acid. His figures show that the range of variation of the nitrogen (13 to 15·5 c.c. per litre, and only 18·5 in a very foul crude sewage) is much less than that of the oxygen (7·5 to nothing), and that the amount of carbonic acid is largely affected by rain.

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* *Proc. Royal Dublin Society*, ix., 3, No. 25, September 3, 1900.

We believe, therefore, in the value and necessity of immediate and also progressive determinations of dissolved oxygen, and we have shown that by our modified Winkler process it is possible to ascertain the amount of free oxygen rapidly in the field in waters so coloured and polluted as to be amenable, as far as we have ascertained, to no other process.

Dr. Rideal also exhibited a graduated bulb apparatus that he had had made, which obviated the use of pipettes.

DISCUSSION.

Mr. DIBDIN said that he had sytematically contended that the degree of aeration was one of the most important factors in determining the quality particularly of sewage effluents, or the character of river water apart from any particular effluent: for the question applied to the examination of rivers as much as to the examination of any particular effluent or water-supply. There was one point as to which he would have liked to have further information, namely, as to how far this method compared with the gasometric process; because, in the course of numerous experiments upon the determination of dissolved oxygen in the water of the river Thames, he had noticed serious discrepancies in the results obtained by various volumetric processes. Naturally, he had not been content to let the matter rest there, but had tried in many ways to overcome the difficulty. Of course, the question of nitrites was almost an obvious one, but he had also had in mind other substances, such as those contained in the water discharged from gasworks, which would affect the question to a greater or less extent. In the case of the Thresh and Schutzenberger processes, he made experiments to ascertain whether it was possible to eliminate the discrepancies by subjecting the samples of water to oxidation by potassium permanganate. He was sorry, however, to say that, as far as his experiments went, the results were not so satisfactory as he had hoped they might be, and consequently he had adhered to the gasometric process for all work in which accuracy was of importance. For the purpose of indicating a degree of aeration of 50 per cent. or over, volumetric processes were undoubtedly satisfactory; but in cases in which the degree of aeration was lower than 50 per cent., results were obtained which were altogether unreliable. If the author could give some results showing how determinations made by this modification of the Winkler process compared with gasometric determinations, a better opportunity would be afforded of judging as to its value. Nevertheless, whether it was absolute or not, it must be admitted that a process which was portable, and could be applied upon the works or upon the river-bank, offered great advantages. There were many such processes, but certainly those referred to in the present paper were to be ranked as among the simplest of any of the kind that he had seen.

Mr. WYNTER BLYTH said that some years previously he had had considerable experience of the Winkler method in examining the rivers of Devonshire, and he had found it extremely convenient. He had, however, been troubled by the presence of organic matter, but not of nitrites, because the waters of the Devonshire rivers were fairly soft, and the quantity of nitrites in them was infinitesimal. He could not conceive anything that would be more advantageous in testing the pollution of rivers than an oxygen process, because one could go down in a boat and compare the condition of the river above and below the point at which a particular effluent was discharged, the results obtained in many cases being very striking. He might perhaps make one criticism on the apparatus now described, namely, that the accurate measurement of the 10 c.c. run out by means of the mark on the tube would necessarily be somewhat difficult, owing to the comparatively large diameter of the tube; and he would suggest that the tube might with advantage be made narrower, so that an accurate reading could be more easily taken.

Dr. RIDEAL, in reference to Mr. Wynter Blyth's suggestion, said that, as a matter of fact, the best plan was to run out the 10 c.c. into a small measuring vessel. In answer to Mr. Dibdin's criticism, he regretted that at present this modification of Winkler's process had not been compared with the gasometric method for very small quantities of dissolved oxygen. It had, however, been satisfactorily ascertained that the method did give very good results as compared with any other volumetric process; and there was no doubt that the addition of the permanganate beforehand, so as to destroy nitrites, gave far more accurate results. Instead of comparing the method by direct experiment with gasometric processes, it had been compared with Roscoe and Lunt's figures for fully aerated water, which were obtained by interpolation between actual determinations by the gasometric process at intervals of 5°. Their figure for pure water at a temperature of 20° C. was 6·28, and that yielded by this process 6·11; while in the presence of 5 parts per 100,000 of nitrous nitrogen the figure obtained by this process was 6·15. Even in the presence of nitrites, ferrous iron and a large quantity of organic matter, this process gave results almost identical with the gasometric figures.

DETECTION AND ESTIMATION OF PRESERVATIVES IN MILK.

By MEREDITH WYNTER BLYTH, B.A., B.Sc.

(*Read at the Meeting, March* 6, 1901.)

THE use of preservatives in milk has made it necessary for those engaged in the examination of dairy products to spend a great amount of time in the search for the various substances used for this purpose. Some chemists are in the habit of looking for preservatives only in those samples which show no signs of decomposition after a definite length of time; but, as more often than not the time that has elapsed since the milk left the cow is unknown, this method is bound to lead to fallacious results, as the state of decomposition of a milk depends not only on the factor of time, but also on the incubation temperature, and the number and nature of the micro-organisms present in the milk.

The decomposition of milk may, however, be made the basis of a test for preservatives if we bring the samples (1) to the same degree of alkalinity, (2) to the same bacterial condition, (3) to the same incubation temperature.

Having made a large number of experiments at different temperatures and under various conditions, I find that the following method gives excellent results, the primary object of the test being to enable analysts to examine easily a large number of samples without a great waste of time:

EXAMINATION FOR PRESERVATIVES.

1. Measure 10 c.c. of each milk into clean, wide test-tubes.

2. Measure 10 c.c. of a sterile milk, known to be free from preservatives, into a test-tube (these control tubes can be kept ready for use).

3. Add to each milk and to the control 2 c.c. of a very strong slightly alkaline solution of litmus. Now examine all the tubes, and if any of them are not the same shade of blue as the control tube, drop in drop by drop a half-normal solution of sodium hydrate until the correct shade of blue is obtained. This will be found unnecessary in the case of most milks, and will only be necessary when the milks are two or three days old; this process must then be done very carefully.

4. Plug all the tubes with cotton-wool, and heat them in a water-bath kept at a temperature of 80° C. for ten minutes.

5. Allow the tubes to cool, and inoculate each, including the control, with ½ c.c. of sour milk in water (½ c.c. milk to 100 c.c. water). Shake the tubes well. Now let the tubes stand for twenty-four hours at any temperature between 15° C. and 22° C., and then examine. If the control tube is not white, or nearly so, they must be allowed to stand for a longer period.

Those tubes which contain preservatives will remain blue or pink, while the tubes containing no preservatives will behave in the same way as the control tubes, becoming quite white.

The length of time the blue or pink colour takes to become white depends upon the quantity of preservative present in the sample.

The quantities of the more common preservatives which can be detected with certainty by this method are 0·005 per cent. of borax, boracic acid, or mixtures of these substances, 0·05 per cent. of salicylic acid, and 0·0003 of formic aldehyde, quantities very much smaller than are ever found, or which would be of any value in commercial milks. Having selected by this method those samples which contain preservatives, the nature of those preservatives must be determined by the ordinary methods.

ESTIMATION OF PRESERVATIVES.

The quantity of any preservative in milk may be estimated by a modification of the process just described. It is not of much use in the case of borax, boracic acid, or salicylic acid, as the usual chemical methods are for these substances of greater accuracy than the method to be described; it may, however, be used even with these substances as a control on the results obtained by chemical estimation.

With formic aldehyde the case is different, the chemical methods for the determination of small quantities of this preservative being very unsatisfactory; and since it has a very high antiseptic value, formic aldehyde is admirably adapted to estimation by bacteriological means. The estimation may be made as follows:

Take two test-tubes, and measure into each tube 10 c.c. of the milk containing formic aldehyde (solutions A). Take 10 c.c. of the original milk, and dilute to 100 c.c. with milk free from preservative; measure out two tubes as before (solutions B).

Take 10 c.c. of solution B, and dilute to 100 c.c. with milk free from preservatives; measure out two tubes as before (solutions C).

Prepare three control tubes of 10 c.c., each containing 0·005 per cent., 0·003 per cent., 0·001 per cent. of formic aldehyde respectively (control A).

Prepare four control tubes of 10 c.c., each containing 0·001 per cent., 0·0008 per cent., 0·0005 per cent., 0·0003 per cent. of formic aldehyde (control B).

Treat all the tubes with litmus, heat to 80° C., cool and inoculate in exactly the same manner as if testing for the presence of preservatives.

Place one tube of solution A, one tube of solution B, one tube of solution C, and the three control solutions A, in the warm incubator at 38° C. for twenty-four hours.

Place all the other tubes in the cool incubator at about 22° C. for twenty-four hours.

From a comparison of the colours of the various tubes after twenty-four hours, a very close estimation can be made of the quantity of preservative originally present in the milk, providing that quantity does not exceed 0·5 per cent.; if it exceeds this number still greater dilutions must be made.

The estimation of formic aldehyde should be made either on the fresh milk or on a portion of the milk which has been partially sterilized at 80° C.; but even in this latter case there seems to be some loss of the aldehyde in twenty-four hours.

It is apparently impossible by this method to get an accurate estimation of formic aldehyde by distilling off the aldehyde and estimating it in the distillate, because the distillate possesses a higher antiseptic value than the formic aldehyde originally placed in the milk will account for; this increase is no doubt due to some distillation product of the milk. Further experiments are being made on this point.

DISCUSSION.

Mr. RICHMOND said that during the reading of the paper a doubt whether all the organisms in milk would be destroyed by heating to 80° C. had passed through his mind, but second thoughts convinced him that the organisms in the quantity of sour milk added were so enormously in excess that the few which might remain could have no practical effect. The method seemed to be an excellent one to enable samples of milk to be sorted with a minimum of trouble, so that only those need be examined which probably contained preservatives. With reference to the author's remark to the effect that the distillate from milk was more antiseptic than the milk itself, it was, he thought, firmly established that when formaldehyde was added to milk it combined with the proteids and gradually disappeared. These proteids had not the same properties as those in the original milk. For instance, when heated with hydrochloric acid they were much more difficult to dissolve than the proteids of the original milk. Now, it was extremely probable that the action of the micro-organisms on the milk containing these altered proteids was not the same as in the case of milk with unaltered proteids, but was in fact likely to result in a greater development of acidity; on distillation, especially in a faintly acid solution, formaldehyde would pass over from the compound, and when the distillate was added to milk would, before combination took place, exert its full antiseptic effect, and this seemed to be a reasonable explanation of what the author had observed. In conjunction with Mr. J. B. P. Harrison he had made some experiments on the rate at which acid developed in milk, and had found that when milk treated with formaldehyde began to go sour it went sour at a very much greater rate than milk not so treated. In some cases it was actually found that, after two or three days, milk treated with formaldehyde contained more acid than untreated milk, while in the earlier stages it contained very much less.

Dr. Rideal remarked that, since the organisms present in milk were not only those which produce souring, a preservative might have an inhibiting effect on organisms other than those which produced a souring effect. To test the presence and estimate the amounts of preservatives by means of a mixed breed of souring organisms would not necessarily give any indication as to the value of the preservatives for preserving milk from other organisms than those of the particular kind used for testing. He believed it was not generally known or generally recognised that, although the lactic-acid-producing organisms were the organisms usually present having a souring effect, organisms of the *Coli* group, which many observers have found in milk, had also this particular property.

Mr. M. Wynter Blyth, in reply, said that his object in heating the milk to a temperature of 80° C. for ten minutes was to kill all organisms except spore-bearing organisms, which in twenty-four hours were not incubated out sufficiently to affect the results. He first of all tried inoculating samples with specific bacilli—*B. coli communis*, and various others—but found that there was no advantage to be obtained in that way over merely inoculating with a very dilute solution of sour milk. With regard to Dr. Rideal's suggestion that the acid-forming organisms could not be used for determining the power of an antiseptic, he (Mr. Blyth) thought that the organisms which decolorized litmus were not necessarily acid-forming organisms, or one would expect the litmus to be turned red. With borax and boracic acid the blue colour disappeared into white without any intermediate red stage, so that there would not seem to be a great number of acid-forming organisms present. The organisms seemed to act on the litmus itself and destroy the colour.

NOTE ON THE APPROXIMATE ESTIMATION OF FORMALDEHYDE IN MILK.

By J. F. Liverseege, F.I.C.

(*Read at the Meeting, March* 6, 1901.)

In September, 1899, I commenced some experiments on the determination of formaldehyde in milk, and, though other work prevented me from continuing the investigation, a short account of my results may be of interest in connection with Mr. M. Wynter Blyth's paper (see preceding paper).

The reagent used was strong pure sulphuric acid, with 2·5 c.c. normal ferric chloride added per 100 c.c., this being the proportion which experiment showed gave the best colour with formalin in milk. Four mixtures were made containing 0·05, 0·5, 5, and 50 parts of formaldehyde per 100,000 of milk. When a small quantity of each milk was floated on to the reagent and allowed to stand all night, the one containing 0·5 gave an intense violet ring, the one containing 50 parts hardly showed any colour, and the other two gave distinct violet rings. Evidently this was of no value for quantitative work.

After trying shaking various quantities of milk with the reagent I arrived at the following method of working: Ten c.c. of each sample of milk were taken, placed in a 25 c.c. stoppered cylinder, and the reagent was added—preferably from a burette—1 c.c. at a time, to each cylinder in turn, and the colour was observed. The larger the proportion of formaldehyde the sooner does a violet colour appear, and on the addition of more reagent the intensity of the colour increases up to a certain point. The following table will illustrate this:

Reagent. c.c.	Formaldehyde present per 100,000.			
	0	0·5	5	50
2nd	0	0	0	Pale violet.
4th	0	0	Pale violet	Intense violet.
6th	Brownish-yellow	Reddish-violet	Intense violet	Violet, paler than 5.

No distinction could be made between milk with 0·05 per 100,000 of formaldehyde and that containing none. Another example in which the colours were quite distinct all through the experiment will suffice:

Reagent. c.c.	Formaldehyde present per 100,000.			
	0	1	5	10
2nd	Yellow	Buff	Violet	Violet.
4th	Yellow	Violet tinge	Violet	Dark violet.

In practice, the acid is added to 10 c.c. of milk side by side with standard samples, and they are compared after the addition of each c.c. of reagent. The standard samples were made from an aqueous solution of formaldehyde prepared by diluting 1 c.c. of formalin with water to 350 c.c., and assaying by the iodometric method (ANALYST, xxii., 221). I may say that a decided excess of iodine is necessary, or the results may be very much too low. The solution was then diluted to contain 1 part of formaldehyde in 10,000. This solution keeps very well.

Whether the quality of the milk makes any difference to the intensity of the colour, and if there is any advantage in cooling the cylinder between the addition of each c.c. of the reagents, are points that require investigation.

SOME ANALYSES OF OATMEAL.

By Bernard Dyer, D.Sc.

Very meagre information is given in easily accessible English books of reference on the subject of oatmeal. In Thorpe's "Dictionary of Applied Chemistry" the average is given of nine analyses by König (chiefly), and also the average of six analyses of American oatmeal by Johnson, from the Report of the Connecticut Agricultural Station for 1888. In the two averages the proportions of albuminoids (nitrogenous substances) are given respectively as 15·0 per cent. and 14·7 per cent., and the proportions of fat as 6·6 per cent. and 7·1 per cent. In Wynter Blyth's "Manual of Foods" the mean composition of oatmeal is given, showing 11·73 per cent. of albuminoids (nitrogenous matter) and 6·04 per cent. of fat. In Church's manual, "Food," an analysis of Scotch oatmeal is given, showing 16·1 per cent. of albuminoids and 10·1 per cent. of fat, and one of Irish oatmeal showing 14·8 per cent. of albuminoids and 8·8 per cent. of fat.

A short time since I happened to have a number of samples of oatmeal submitted to me for analysis as to genuineness under the Sale of Food and Drugs Act, and I took the opportunity of making a proximate analysis of each, thinking that the results might possibly be useful to myself or to others for subsequent purposes of reference, and in pursuance of this idea I have brought the results together in the following table.

I may mention that the figures given in the column headed "Albuminoids" are obtained by multiplying the percentages of total nitrogen by 6·25, and do not express true albuminoids as distinguished from other nitrogenous bodies.

The indigestible fibre is obtained in the way ordinarily employed in the analysis of feeding stuffs, and the figures in the column headed "Starch and other Digestible Carbohydrates" are obtained merely by "difference."

Thirteen of the samples were what would in common language be described as "fine oatmeal," six were of various grades that would ordinarily be described as "coarse oatmeal," while eight samples represented proprietary preparations in common use, consisting of crushed or flattened oats rather than of what is ordinarily described as oatmeal.

It will be seen that the albuminoids, calculated by multiplying the percentage of nitrogen by 6·25, varied from about 13 to over 18 per cent. In ten cases the proportion of albuminoids did not exceed 14 per cent.; in ten cases it was from 14 to 15 per cent.; in five cases from 15 to 16 per cent.; and in two cases over 18 per cent. In the two cases in which the albuminoids reached 18 per cent., the proportions both of ash and indigestible fibre will be seen to be large, as though the proportion of farinaceous substance to integument was smaller in these cases.

As regards oil or fat, it will be seen that in no case did the proportion fall as low as 7 per cent. In five cases it was between 7 and 8 per cent.; in eight cases between 8 and 9 per cent.; in ten cases from 9 to 10 per cent.; in two cases between 10 and 11 per cent.; and in two cases a little over 12 per cent.

	Moisture.	Oil.	Albuminoids.	Starch and other Digestible Carbohydrates.	Indigestible Fibre.	Total Ash.	Ash insoluble in Dilute Hydrochloric Acid.	Nitrogen.
Fine oatmeal ...	9·07	9·53	16·00	60·86	2·07	2·47	·17	2·56
,, ,,	9·10	8·95	14·81	63·71	1·50	1·93	·07	2·37
,, ,,	9·53	8·63	13·06	65·65	1·30	1·83	·07	2·09
,, ,,	9·17	9·17	13·62	64·94	1·20	1·90	·17	2·18
,, ,,	9·00	9·23	12·94	65·76	1·10	1·97	·10	2·07
,, ,,	8·83	12·23	18·19	54·65	2·20	3·90	·10	2·91
,, ,,	9·00	8·60	14·00	65·46	1·17	1·77	·10	2·24
,, ,,	8·27	10·63	14·44	62·79	1·70	2·17	·13	2·31
,, ,,	9·10	9·97	13·94	63·16	1·70	2·13	·13	2·23
,, ,,	8·60	12·33	18·44	54·40	2·20	4·03	·27	2·95
,, ,,	8·83	8·67	13·12	66·31	1·20	1·87	—	2·10
,, ,,	8·20	9·70	15·31	62·09	1·80	2·90	·17	2·45
,, ,,	8·33	9·37	14·93	63·81	1·43	2·13	·03	2·39
Coarse oatmeal	7·97	8·77	15·18	65·15	1·03	1·90	·07	2·43
,, ,,	9·00	10·23	13·06	65·07	·87	1·77	·10	2·09
,, ,,	8·33	9·07	14·31	65·25	1·07	1·97	·07	2·29
,, ,,	8·43	8·73	15·46	64·51	1·07	1·80	·07	2·49
,, ,,	7·93	9·00	14·93	65·04	1·13	1·97	·10	2·39
,, ,,	9·17	9·37	13·69	64·84	1·13	1·80	·07	2·19
Crushed or flattened oats ...	8·70	7·57	14·93	66·13	·97	1·70	·10	2·39
,, ,,	8·17	7·80	14·44	66·82	·87	1·90	·10	2·31
,, ,,	8·57	7·87	13·94	67·02	·87	1·73	·07	2·23
,, ,,	8·93	8·43	14·44	65·50	·87	1·83	·07	2·31
,, ,,	9·03	7·63	14·56	66·21	·80	1·77	·07	2·33
,, ,,	9·27	9·30	12·68	66·21	·87	1·67	·07	2·03
,, ,,	8·77	8·17	15·18	65·24	·87	1·77	·07	2·43
,, ,,	8·60	7·53	14·81	66·12	1·17	1·77	·07	2·37

The "fibre" showed variations from 0·80 to 2·20 per cent., and the ash from 1·67 to 4·03 per cent.

DISCUSSION.

Mr. LING inquired whether any determinations had been made of the furfural-yielding carbohydrates.

Mr. CHAPMAN (Hon. Sec.), in the absence of the author, said that the carbohydrates appeared to be represented by the two items "indigestible fibre" and "digestible carbohydrates" (the latter of which was obtained by difference), no further differentiation of the carbohydrates having been made.

Mr. DIBDIN inquired whether all of the samples were genuine. So high a proportion of albuminoids as 18 per cent. seemed to point to some abnormality.

Mr. CHAPMAN said he was informed that all the samples were genuine.

Mr. HEHNER said that, in examining a specimen of one of the largely advertised preparations of crushed oats, he had found, rather to his surprise, that while the fat of oats and of fine or coarse oatmeal was largely composed of free fatty acid (as in the case of finely crushed seeds containing fat), the fat in this particular kind of oats contained practically no free fatty acid. The material had evidently undergone some process of treatment to neutralize the free fatty acid, somewhat in the same way in which cocoa was treated with ammonia to render it soluble; and this might possibly account for the high nitrogen contents of one or two of the samples referred to in the paper.

The PRESIDENT (Dr. VOELCKER) said it was certainly of great advantage that figures of this kind should be collected, so that there would not be the necessity of referring always to foreign authorities. At the same time, he had often felt that the mere collection of analytical results of food materials, without definite information as to the source from which the materials came, or of the circumstances under which they were produced, was likely to lead to a great deal of misapprehension. It was most valuable to have extended investigation into samples that had been produced in particular ways and under known conditions. If, for instance, in the present case information were available as to whether the terms "fine oatmeal," "coarse oatmeal," and "crushed or flattened oats," were simply names that happened to be put upon the samples in the course of collection, or whether they represented intrinsic differences between the different materials, it would enable some conclusion to be drawn as to how the average composition altered according to the more or less thorough removal of different portions of the grain. It was well known that in wheat, barley, or other cereals the removal of the different layers which covered the inner part of the grain made an enormous difference in the percentage of fat, and also in the nitrogenous constituents of the grain. The question of whether grain was highly nitrogenous or highly starchy depended upon two main conditions. The first was what was known as "maturation," or, in other words, the condition of things at the time of ripening—whether the grain had ripened rapidly, or whether the development and laying up of starch had been a gradual process. The second factor—a factor which, he thought, exercised more influence than it had yet been credited with—was the kind of soil upon which the particular grain had been grown. From the experience that he had acquired within the last two or three years in the growing of grain under different conditions, he was convinced that in the case, for instance, of wheat grown on the same soil and from the same variety of seed, hard wheat or soft wheat might be obtained according as the season was a rapid ripening one or one of mild and steady growth. If the weather were very hot just at the time of ripening, the plant was highly nitrogenous, and a "steely" grain was the result; whereas if the ripening were gradual, a more starchy and less nitrogenous grain would be obtained. Then, again, in growing the same seed—wheat, for instance—on two different soils, it might almost be said that it was possible to transform a soft wheat into a hard one, and *vice versâ*. Such factors as these, therefore, must influence very much the value of tables of analytical figures, and render desirable more definite information as to what the particular products are, how they have been produced, and what is the influence of the surrounding conditions in altering their composition.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

The Estimation of Fat in Sweetened Condensed Milk by the Babcock Test. E. H. Farrington. (*Amer. Chem. Journ.*, xxiv., 267.)—When the Babcock test is applied to the estimation of fat in condensed milk which has been sweetened by sugar, a black flocculent substance separates in the neck of the test-bottle, and a satisfactory reading cannot be obtained. Extraction of the fat with ether is also unreliable. In such cases the following method, in which the sugar is first removed, has given good results. Sixty grammes of the condensed milk are weighed into a 200 c.c. graduated flask, the volume is made up to the mark with water, and 17·6 c.c. transferred to a Babcock test-bottle by means of a pipette. About 3 c.c. of the sulphuric acid used for testing milk are added, and, after mixing well, the bottle is whirled for six minutes in a centrifuge heated to 200° F. by means of steam; this causes the curd to shrink into a firm mass. If the curd is not broken, nearly all the whey, containing the sugar, can be decanted off. The curd is then shaken up with 10 c.c. water, and the whirling repeated after the addition of another 3 c.c. acid. The whey is again decanted, the curd shaken up with 10 c.c. water, 17·5 c.c. sulphuric acid added, and the test completed in the usual way. The small amount of sugar left does not interfere with the testing.

A. G. L.

The Occurrence of Methyl Alcohol in Fermented Fruit Juices. J. Wolff. (*Zeit. Untersuch. Nahr. Genussm.*, 1901, iv., 391-394.)—The author has examined the juices of various fruits before and after fermentation. With the exception of black currants, which yielded a distillate containing a trace, no methyl alcohol was found in any of the juices before fermentation. The distillates from the fermented juices were tested by Trillat's method (ANALYST, xxiv., 13, 212), and the following proportions by volume were found, calculated on 100 parts of 90 per cent. alcohol:

		Per Cent.
Black currants		Above 2
Plums		About 1
Damsons		„ 1
Mirabelles		„ 1
Cherries		0·5 to 1·0
Apples		0·2 „ 0·3
Grapes,	without stalks	trace „ 0·03
	with stalks	0·15 „ 0·4
	skins	0·15 „ 0·6

White crystalline sugar fermented with wine yeast produced no methyl alcohol. It is interesting to note that the brandy prepared from "marcs" invariably contains an appreciable quantity of methyl alcohol, whilst good cognac only gives uncertain indications in this test.

Rums, whiskies and commercial alcohols of different origin were found free from methyl alcohol. In the application of Trillat's test the following rules were observed:

1. In testing brandies, the quantity of liquid used corresponded to one containing 10 c.c. of 90 to 95 per cent. alcohol.

2. Blank tests were made with 10 c.c. of pure 90 to 95 per cent. alcohol, and tests with the same amount of alcohol containing 0·02 and 0·05 c.c. of methyl alcohol.

C. A. M.

Estimation of Total Extract and Free Acid in Wine. F. Freyer. (*Oesterr. Chem. Zeit.*, 1901, iv., 129.)—In view of the many possible sources of error in the usual process of determining the total extract in wines by evaporation, the author suggests that it would be better in all cases to arrive at this figure by taking the specific gravity of the sample before and after distillation of the alcohol, using either the Haas or the Windisch tables for the necessary calculations.

As regards the "free acid" in wine, Freyer remarks that the amount of alkali required for neutralization depends upon the presence of a large number of different acids, not all of which are yet accurately known; whereas the loss of acidity which wine undergoes on storage is almost entirely due to the separation of potassium bitartrate. But since 188 parts of tartar are equal to only 75 parts of free acid, the total extract decreases out of proportion to the loss of acidity of a wine as tartar is eliminated; so that the acid-free extract, which is an important factor in judging wine, is made to appear lower after separation of tartar than before. It is therefore desirable to make a special determination of the potassium bitartrate in the wine, to calculate this into its equivalent of free tartaric acid (188 = 75), to deduct the value so obtained from the total non-volatile acid by titration, and to return the difference as true free acid. The non-acid extract is then given by subtracting both the tartar and the corrected free acid from the total extract.

F. H. L.

Detection of Arsenic in Beer. (*Chemical News*, vol. lxxxii., pp. 302, 303; vol. lxxxiii., pp. 2, 22, 25, 26.)—A. W. Blyth (vol. lxxxii., p. 302) eliminates alcohol and carbon dioxide by boiling, adds caustic soda till alkaline, evolves hydrogen from sheet aluminium, and passes the gas through 1 c.c. of silver nitrate solution. This method will detect 0·01 milligramme of arsenious acid in 100 c.c. of beer, and is not interfered with by the presence of acid sulphites.

H. G. Madan (vol. lxxxii., p. 302) recommends a modification of Fleitmann's test: heating 6 to 8 c.c. of the beer, etc., to near boiling with 1 gramme of caustic potash, dropping a piece of aluminium foil into the solution, and applying to the mouth of the tube a strip of filter paper moistened with silver nitrate, which will blacken if arsenic be present. Sulphur and phosphorus, being detained by the alkaline liquid, do not interfere with the test.

J. S. Smith (vol. lxxxiii., p. 2) finds that arsenic can be detected, in the presence of sulphur compounds that give off sulphuretted hydrogen (on treatment with zinc and acid), by heating the evolved gases in Marsh's apparatus with the following precautions: Adding the solution to the zinc and acid by 1 to 2 c.c. at a time;

keeping the heated tube red hot for about 1 inch in the centre, to prevent loss; washing the cooled deposit with carbon disulphide, to remove sulphur, and afterwards with caustic alkali, to dissolve the arsenic sulphide. The final rinsings with water are added to the solution, and the whole is neutralized with hydrochloric acid, the precipitate being tested by the usual methods. Where the sulphur is insufficient to combine with all the arsenic, a metallic ring of the latter will form beyond the deposit of sulphide.

J. W. Gatehouse (vol. lxxxiii., p. 22) states that the method proposed by Madan (see above) was described by himself in 1873 (*Chemical News*, April 18). It is necessary to employ pure caustic alkali, some commercial qualities containing arsenic.

E. W. T. Jones (vol. lxxxiii., pp. 25, 26) recommends, as a *qualitative* test, concentrating 250 c.c. of the beer to about 100 c.c., adding 25 c.c. of strong hydrochloric acid, and immersing a strip of clean copper gauze in the boiling liquid for a quarter of an hour. If this be darkened, it is then heated in a (preferably elliptical) tube, and the sublimate examined under the microscope; crystals of arsenious oxide will be readily detected when $\frac{1}{20}$ grain per gallon is present. For glucose, etc., 50 grammes are dissolved to 100 c.c. in hot water, and treated in the same way.

In the *quantitative* test, two rolls of copper gauze (1 by $3\frac{1}{2}$ inches) are used alternately, the first being removed, after an hour's boiling, and freed from adherent deposit by 5 c.c. N. caustic soda (diluted to just cover the copper in a small beaker), and 3 to 4 drops of a 10 volume solution of hydrogen peroxide. The second roll is boiled for half an hour, and then treated as above, the process being repeated till no further deposit shows. The alkaline solution and washings are heated for an hour, filtered from the precipitated copper, and treated with 7 c.c. of normal sulphuric acid and a little pure sulphurous acid solution; then boiled to expel all traces of the latter, and treated with its own volume of saturated sulphuretted hydrogen solution, followed by gaseous H_2S. After standing over-night in a warm place, the deposited arsenious sulphide is filtered, thoroughly washed, dissolved in a minimum of hot dilute ammonia water, evaporated to dryness, taken up (when cool) with 2 to 3 drops of hot water, and slightly acidified with very dilute hydrochloric acid. It is then evaporated to dryness, treated with 2 to 3 drops of H_2S water, again evaporated, washed with a little water, dried, freed from sulphur by carbon disulphide, rinsed with strong alcohol followed by water, thoroughly dried, cooled, and carefully weighed. The arsenious sulphide is then dissolved with hot ammonia water, and the residual copper sulphide is dried and weighed, the difference giving the weight of arsenious sulphide present. The method will isolate 0·9 out of 1 grain of arsenious oxide added per 1 gallon of non-arsenical beer.

C. S.

New Therapeutic Agents of the Year 1900. A. Eichengrun. (*Zeits. angew. Chem.*, 1901, xiv., 261.)—I. (*a*) ANTISEPTICS FOR EXTERNAL USE: *Iodolen* (Laquer), a substance of doubtful value, is a compound of iodol (Squire, p. 373[*]) with albumin. *Iodylin* (Stephan) is an iodine derivative of unknown composition. *Iodozen* (Merrel) is iodized oil of winter-green, an American substitute for sanoform (p. 313). *Vioform*, made by the Basle chemical works, is iodochloroxyquinoline, and is advertised as a substitute for iodoform. *Proteol*, a secret preparation recommended in the same way, is presumably analogous to the compounds of formaldehyde and albumin. *Airogen*, like *airoform*, is simply an imitation of airol (p. 145). *Ibit* (Ges. Chem. Ind., Basle) is bismuth oxyiodotannate. *Crurin* (Edinger) is bismuth quinoline thiocyanate; commercially it varies somewhat in composition. *Marcasol* (Kress and Owen) is bismuth borate and phenoxide. *Mercurol* (Schwickerath) is the mercury salt of nucleic acid. *Cuprol* is the similar copper compound, and *cupragol* (F. Bayer and Co.) is a compound of copper with proteic acid; but these are still in the experimental stage. *Nargol* is the silver salt of nucleic acid, and like the preparations argonin and protargol (p. 113), as well as those mentioned above, contains the metal firmly united to the organic molecule. *Ichthargan*, a compound of ichthyol and silver, on the other hand, is readily split up by hydrochloric acid and chlorides. *Ichthoform* is a derivative of formaldehyde and ichthyol sulphonic acid. The various solutions of phenols in ichthyol, formerly called *anytols*, have now been given special names, and include *metasol* for *m*-cresol-anytol, and *eucasol* for eucalyptus-anytol. This latter designation is unfortunately liable to cause confusion with the food-stuff eucasin. As competitors with ichthyol are now proposed, *ichthydrin*, a by-product in manufacturing the former; *illyrin*; *sphagnol*, or *corba oil*, a black tarry substance distilled from peat; and *Pix solubilis*, a soluble modification of the tar obtained by sulphonation.

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[*] References to the seventeenth (1899) edition of Squire's "Companion to the British Pharmacopœia" have been interpolated into this article by the abstractor to save space.—F. H. L.

I. (*b*) ANTISEPTICS FOR INTERNAL USE.—*Bromeigon* (Dieterich) and *iodeigon* are halogen derivatives of peptone (if soluble), or of albumin (if insoluble). *Bromocoll* (Brat) is brominated tanocoll (*i.e.*, gelatin-tannin). *Guaiakinol* (Castell) is the dibromoguaiacolate of quinine, or perhaps a compound of the formula

$$C_{20}H_{24}N_2O_2.2HBr.C_6H_4OH.OCH_3.$$

A body called "*eosolsaures Salz*" (Wendt) is very similar to thiocoll, and is probably a salt of acetylcreosotetrisulphonic acid. *Guaiamar* (Endemann) is the glycerol ester of guaiacol. *Guaiasanol* (Einhorn) is diethylglycocollguaiacol:

$$C_6H_4.OCH_3.OOC.CH_2.N(C_2H_5)_2.$$

Fluoroform is made, according to Meslans, by the action of silver fluoride upon iodoform in aqueous solution. *Igazol* (Cervello) is stated to be a "compound" of formaldehyde with trioxymethylene and some iodine derivative; but the author finds it to be simply a mixture of paraform, iodoform and terpene hydrate. *Aniodol* (Sédan) is a solution of paraform in glycerol mixed with oil of mustard; but owing to the decomposition which occurs on dissolution is really a weak formaldehyde solution. *Lysoform* is a scented solution of formaldehyde in soap, much like lysol. *Phenolysolum hungaricum* and *basol* are merely new names for lysol. *Negrolin* and *kresosolvin* are new names for creolin; *saprol* is apparently something similar. *Pictolin* is a mixture of liquefied carbon dioxide and sulphurous oxide.

Two new substances are *epicarin* (Bayer), β-naphthol cresotinic acid, and *peruol* (Actienges. für Anilinfabr.), recognised by E. Erdmann as the active constituent of Peru balsam. The latter, dissolved in castor-oil, is called *peruscabin*. *Sapodermin* and *lavodermin* are soap preparations containing about 3 per cent. of the mercury compound of casein. *Viscin* is birdlime (from the white mistletoe). *Chirol* is a solution of resins and fatty oils in ether-alcohol.

II. NEW DRUGS.—Two new hypnotics are *hedonal* (Beyer),

$$NH_2CO.O.HC.CH_3.C_3H_7,$$

and *chloretone* (Parke Davis), which is simply acetonechloroform. Under the name of *anesone* acetonechloroform has been known for years as an anæsthetic. *Mannin* is the title given to an imitation of orthoform. Of antipyretics, *eupyrin* (Zimmer) is the ethylcarbonate of the condensation product of vanillin with phenetidine prepared by Goldschmidt. Others are acetylsalicylic acid, and the compound of pyramidone with camphoric acid (Höchst). *Acetopyrin* is the name given to the compound of antipyrine with acetylsalicylic acid; it is very unstable, losing the acetyl group. *Sidonal* (Jaffé and Darmstädter) is piperazine quinate, and *urosin* is lithium quinate. It seems questionable whether the effect of sidonal is not due to the piperazine, which is thus exhibited in unusually large doses; nevertheless, many other quinates are now being patented. *Kineurin* is quinine glycerophosphate. *Euchininum tannicum* is a perfectly tasteless gallate of euquinine. *Basicin* (Kreidmann) is a mixture of quinine hydrochloride and caffeine. *Yohimbine* is an alkaloid obtained from a West African plant. *Fortoin* (Zimmer and Co.) is the methylene compound of cotoin (Squire, p. 246). *Honthin* (v. Sztankay) is a Hungarian variant of tannalbin (p. 52), and is claimed to be an improvement. *Lactanin* (Schmoll) is bismuth lactotannate, prepared by acting upon bismuth tannate with lactic acid. *Bismuthan* (Kalle and Co.) is a combination of bismuth with albumin. *Ko-sam* is an extract of the Brucea Sumatrana. *Cascarin*, the active principle of the laxative cascara sagrada, has now been introduced commercially. *Ricinose* is merely a new name for castor-oil. *Gomenol* (Prevet and Co.) is a preparation of some New Caledonian plant. *Kalagua* is a Belgian tonic containing caffeine, presumably derived from the *Theobroma Kalagua*, a variant of the Cacao.

Furunculin, *staphylase*, *mycodermine*, *sitogen* and *force* are new malt preparations. *Buffo* is a dilute form of *toril*, a variety of beef extract. *Nervin* is a new extract of meat. *Zômol* is a soluble solid meat-paste. *Nectrianin* is a French product from the *Nectria ditissima* of apple and pear trees.

Tuberculin soap is a new preparation for external use. Behring and Ruppel have isolated free *tuberculic acid* from its compound with tuberculosamine by treatment of the cultures with acetic acid; this substance is said to retain its activity without addition of glycerol longer than any others. *Tuberculol* is the name of an extract obtained by the fractional extraction at increasing temperatures of the tubercle cultures, and is reported to be more efficient, as the heat of the old process destroyed certain principles.

Pegnin is a rennet preparation which yields as finely divided a curd from cows' milk as from human milk. *Pankreon* (Thomas and Weber) is a preparation of the pancreas of high albumin-dissolving power, and capable of withstanding the stomach pepsin for a long time. *Robol* is an amylolytic and proteolytic ferment of secret character. *Roborin* and *roborat* are probably somewhat similar.

The mixture of methyl and ethyl chloride used for producing local anæsthesia is now called *kelenmethyl*, *anesthyle*, *metaethyl* and *anesthol*; orthoform is known as *mannin*; migranin is called *phenazon*. *Sycorin*, *glycosin* and *glycophenol* are fresh names for saccharin; and its methyl ester is called *amerol* in America (probably for tariff reasons, as it is hydrolyzed locally). Theurer's compound of albumin and hæmoglobin is called *perdynamine*; the starch syrup nutrol is now known as *nural*; and *mercurial* has been changed to *mercuralgam*. Salts of cacodylic acid are being recommended in France in general therapeutics under the names of *arsykodyls*. *Persodine* is a mixture of ammonium and potassium persulphate. *Diathesin* is a name for saligenin. *Cystamin* (Squire and Sons) is hexamethylenetetramine; *cystogen* is an American name for the same. *Calmin* is a mixture of antipyrine and heroin; *antirhinol*, a mixture of tannin, salol and sandal oil. *Vitafer*, largely advertised as magnesium peroxide, is a mixture of magnesium carbonate and sodium sulphate, and contains only a trace of active oxygen. F. H. L.

Notes on Phosphorated Oil. K. Stich. (*Wiener klin. Wochenschr.*, 1901, xiv., 177; through *Chem. Zeit. Rep.*, 1901, 96.)—The author finds that the limit of delicacy of Mitscherlich's test for phosphorus in oil is 0·2 milligramme per 100 grammes. Light, air, and rancidity appear of small influence upon the permanence of weak solutions, and this qualitative test succeeds equally when the samples are several weeks old. Quantitative results cannot be obtained by oxidizing the steam-distillates from phosphorated oil. It is better to dissolve the oil in benzene, precipitating with acetone and silver solution, and oxidizing the silver phosphide. This process should not be attempted for qualitative purposes, since cod-liver oil (without phosphorus) gives a black precipitate with the same reagents. Phosphorated oil stored in half-filled bottles yields a yellow amorphous deposit, partly consisting of a special modification of amorphous phosphorus, and partly of some product of imperfect oxidation. F. H. L.

[*Cf.* ANALYST, 1899, xxiv., 91.]

TOXICOLOGICAL ANALYSIS.

Is the Blondlot-Dusart Method reliable in Medico-Legal Cases? Z. Halász. (*Zeit. anorg. Chem.*, xxvi., 438.)—Considerable doubt has been thrown on the reliability of this method for the detection of phosphorus since the publication of Selmi's dissertation on the occurrence of phosphorus bases in the urine and various organs in cases of acute phosphorus poisoning (*Archiv. d. Pharm.*, 1881, xix., 276). In the one case examined which had ended fatally, Selmi was able to obtain from the brain and liver three bases containing phosphorus, which evolved phosphoretted hydrogen in contact with nascent hydrogen. Consequently he suggested that the examination of the brain and liver might be of great importance in cases where it is impossible to detect free phosphorus, since the normal constituents of the brain containing phosphorus are derived from phosphoric acid, which is not reduced under any circumstances by nascent hydrogen.

In three works dealing with the subject which have appeared since Selmi's dissertation, reference is made to his work, and, his results evidently being misinterpreted, the Blondlot-Dusart method is stated to be unreliable. The author of the present paper has consequently investigated the question. As materials for his experiments he used fresh and more or less decomposed human brains, and brains of pigs and calves which had not been poisoned, and also the brains and other organs of rabbits poisoned in different ways by means of phosphorus. In each case the organ to be examined was macerated and mixed into a paste with recently boiled water. This paste was then treated with nascent hydrogen, obtained from zinc and sulphuric acid, in a flask heated on the water-bath. The gas was evolved for two hours, and passed through a large Pettenkofer tube containing silver nitrate. The resulting precipitate was filtered off, washed, and examined in a Dusart apparatus. This consists of a flask in which hydrogen is evolved, connected with an empty wash-bottle to retain moisture, leading to two U-tubes filled with pieces of potash, from which proceeds a glass tube terminating in a platinum nozzle, the gas being lit at the nozzle and examined in a dark room.

In no case was the green coloration of the inner zone of the flame which constitutes the Blondlot-Dusart reaction obtained when non-poisoned human brains and brains of pigs and calves in different stages of decomposition were examined in this way; nor could any phosphorus compounds volatile with water or alcohol be obtained from fresh or decomposed non-poisoned human brains. On the other hand, phosphorus compounds volatile with alcohol were found in the brains of rabbits poisoned by means of phosphorus, although these never showed the Blondlot-Dusart reaction. This reaction, however, was given by other organs, especially the stomach and intestines, as well as the liver, lungs and kidneys. It seems probable that before the phosphorus can reach the brain it is already in too oxidized a condition to give the reaction.

From his experiments the author concludes that the Blondlot-Dusart method may be used with perfect confidence in cases of phosphorus-poisoning. A. G. L.

Differentiation of the Blood of Different Animals. S. Cotton. (*Bull. Soc. Chim.*, 1901, xxv., 255-257.)—According to the results of the author's experiments, the blood of different animals varies in intensity in its action upon hydrogen peroxide, and on this he bases the following test: The fresh warm blood is expressed in a linen cloth until only the fibrin remains. The expressed liquid is shaken until homogeneous. One c.c. is then treated with 250 c.c. of hydrogen peroxide (12 vols.), and the liberated oxygen collected and measured.

In this way the following results were obtained:

	C.c.
Man	580 to 610
Horse	320 „ 350
Pig	320 „ 350
Ox	165 „ 170
Guinea-pig	115 „ 125
Sheep	60 „ 65

The blood of female and young animals gives somewhat higher results than that of old male animals. C. A. M.

ORGANIC ANALYSIS.

Constants of Lard Oil. M. Duyk. (*Bull. de l'Ass. belge*, 1901, xv., 18, 19.)—The author gives the following results which he has obtained in the examination of lard oil: Solidification point about 10° C.; specific gravity at 14° C., 0·916; at 100° C., 0·8626; refractive index (Zeiss) at 40° C. = 52°; critical temperature (Crismer) in an open tube = 75° C.; Maumené figure (50 grammes of oil + 10 c.c. of sulphuric acid of 1·88 specific gravity) = 47° C.; iodine value (mean), 73; elaidin reaction; hard mass in a short time. Saponification value, 193; insoluble fatty acids, 97·4 per cent.; volatile fatty acids, 0.

The fatty acids melted at 35° C., solidified at 31° C., and had a specific gravity at 100° C. of 0·885. C. A. M.

Oil of Walnut (Juglans nigra). L. F. Kebler. (*Amer. Journ. Pharm.*, 1901, lxxiii., 173, 174.)—The author states that he has prepared this by cold expression from the crushed kernels, the yield being 25 per cent., whilst the kernels contained 66 per cent. of oil.

The oil thus extracted was limpid and of a straw colour. It became turbid at −12° C., and had the following physical and chemical constants:

Specific gravity at 15° C.	0·9215
Saponification value	190·1 to 191·5
Acid value	8·6 „ 9
Ester value	181·5 „ 182·5
Hehner value	93·77
Reichert-Meissl value	15 c.c.
Iodine value	141·4 to 142·7
Melting-point of fatty acids	0° C.

Its drying properties were found to be equal, if not superior, to those of linseed oil. A flexible transparent film was obtained which was less liable to crack than the linseed oil film. C. A. M.

Analysis of Oils containing Carvone. E. Kremers. (*Journ. Soc. Chem. Ind.*, 1901, xx., 16.)—This article consists essentially of a résumé of several papers already published by Kremers and his collaborators upon the determination of carvone as carvoxime.* The process is as follows: A quantity of the oil to be analysed, containing about 5 grammes of carvone, is dissolved in 25 c.c. of alcohol, and 5 grammes of hydroxylamine hydrochloride and 6·5 grammes of sodium bicarbonate are added. The mixture is boiled for half an hour on the water-bath under an inverted condenser, 25 c.c. of water are introduced, and the alcohol is distilled off. Steam is then passed through the flask and the distillation is continued, collecting the last portions of the distillate in separate test-tubes till traces of carvoxime crystals appear on the surface of the liquid. The distillation is stopped, the condenser rinsed with a little hot water, which, together with the last distillates, is returned to the still. The whole is cooled, and when the oxime has solidified it is collected on a filter (removing what adheres to the flask by means of a loop of stiff wire), washed, and dried by suction. The product is finally heated for one hour on the water-bath and weighed. To the weight thus obtained 0·1 gramme is added to compensate for the loss by volatilization, and the carvoxime so corrected is calculated into carvone by the factor 0·9088. It is desirable not to use more sodium bicarbonate than is equivalent to the hydroxylamine hydrochloride, while a large excess of the latter should also be avoided. The volatility of the oxime in presence of water vapour is somewhat irregular, therefore the drying should always be performed under similar conditions.

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* The previous articles will be found in *Journ. Chem. Soc.*, Abstracts, 1898, liv. [ii.], 358; 1900, lviii. [ii.], 117 and 631.—F. H. L.

When oils are much resinified, the carvoxime may refuse to crystallize; in such case the oil should be distilled with steam, and the process carried out on the distillate. A small quantity of resin will increase the weight of oxime recovered, and so tend to make the results too high. The melting-point of the carvoxime, which should lie between 71° and 73° C., is lowered by the resin; but no idea of the amount of impurity can be obtained in this way, as the point at which melting begins in the impure oxime is not sharp. The point of complete liquefaction is more distinct, but it is not so much affected by the resinous matter. Copious tables are included in the original, which tend to show that the results are usually about 2 per cent. too low. The author considers that the method is by no means perfect, but is of much value; and it has the advantage of yielding a definite crystalline compound.

F. H. L.

Detection of Succinic Acid. C. Neuberg. (*Zeits. physiol. Chem.*, 1901, xxxi., 574; through *Chem. Zeit. Rep.*, 1901, 95.)—The author makes use of the pyrrol reaction, which enables him to detect 0·6 milligramme of succinic acid. The liquid to be examined, containing the succinic acid in the free state, is mixed with some ammonia and evaporated to a volume of about 1 c.c., absorbed in 1 gramme of powdered zinc, and the excess of ammonia gently driven off. Deal shavings moistened with strong hydrochloric acid are then suspended in the tube, which is heated till a pale or dark red colour appears on the wood, according to the amount of succinic acid taken. If the latter originally existed as a metallic salt, the same test will frequently succeed when ammonium carbonate is used in place of ammonia; or, to be quite certain, a few crystals of ammonium phosphate may be added to the concentrated ammoniacal liquor before introducing the zinc dust. The reaction is extremely delicate, and quite characteristic provided no substances capable of yielding pyrrol are present; in physiological work the only bodies which have to be considered are albumin, hæmin, and indol derivatives. These, however, can be easily separated from the succinic acid.

F. H. L.

INORGANIC ANALYSIS.

A New Volumetric Method for the Determination of Silver. Launcelot W. Andrews. (*Amer. Chem. Journ.*, xxiv., 256.)—The method is founded on that of Pisani, and is especially applicable to residual analyses of chlorides, bromides, etc., and to their estimation in water analysis. Owing to the powerfully oxidizing properties exhibited during the reaction in the original method, no distinct end-point could be observed, and in order to remedy this a ferrous salt is added, and also an equal amount of a ferric salt, the effect of the latter being to preserve a condition of equilibrium between the ferrous and ferric salts and iodine and iodide, as otherwise the ferrous salt would decolorize the starch iodide. The titration is carried out by means of $\frac{N}{20}$, $\frac{N}{100}$, or even weaker solutions of starch iodide, standardized against a $\frac{N}{10}$ solution of pure silver.

The starch iodide is prepared by grinding up 50 grammes starch with 8 to 9 grammes iodine, stirring the powder with 100 c.c. water, and heating the mixture for several hours in sealed tubes immersed in the water-bath, or in a flask fitted with a reflux condenser. The liquid obtained is diluted and filtered; it does not change on keeping. The silver solution to be titrated should contain nitric acid, but not more than 5 per cent., otherwise the ferrous salt would be oxidized. Mercury arsenious and antimonious salts must be absent, and nitrous acid should be removed by boiling; but phosphates, lead salts, and sulphurous acid are harmless.

To the silver solution so much ferrous salt is added that at least as much iron as silver is present, and then an equal quantity of a ferric salt. An excess of the latter must be added if sulphurous acid is present. The iron solutions are best kept on hand as sulphates, which just before use are converted into nitrates by the addition of lead or strontium nitrate. This is unnecessary if the silver solution contains less than 20 milligrammes Ag per litre, as no silver sulphate would crystallize out in that case. The starch iodide solution is then run in, not too rapidly, until the solution is coloured blue.

Starch iodide reacts with silver chloride, bromide, etc., and consequently, if larger quantities of silver are first precipitated with $\frac{N}{10}$ KBr, the solution must be filtered before finishing the titration with $\frac{N}{100}$ starch iodide. A. G. L.

Rapid Estimation of Small Quantities of Arsenic. A. Atterberg. (*Chem. Zeit.*, 1901, xxv., 264.)—The following method has been elaborated to avoid the use of gas generating apparatus, which are not always at hand when required. It depends on the fact that arsenious acid, when heated with strong hydrochloric acid, yields arsenious chloride, which can be completely condensed in water. On adding nitric acid and evaporating, arsenic acid is left, which in quantities of 0·01 milligramme or less can be detected by appropriate reagents—*e.g.*, molybdate, thiacetic acid, or hydrochloric acid solutions of stannous chloride and sodium hypophosphite. The organic matter of the sample (carpet, fabric, etc.) is destroyed by heating with 50 to 100 c.c. of 1·19 hydrochloric acid, 2 grammes of ferrous sulphate to reduce any arsenic acid, and, in the case of "bronze" (metallic) colours, 2 or 3 grammes of ferric chloride to dissolve any elementary arsenic. The vapours are led through a doubly bent tube into a 50 c.c. pipette, which stands with its lower end under 50 c.c. of water in a 100 c.c. flask, kept constantly cold. By the time the pipette has become hot, practically all the arsenic has distilled over, and the distillate is diluted with water to 100 c.c. Three c.c. thereof are then evaporated to dryness in a round-bottomed basin with 1 c.c. of 1·2 nitric acid; and when the acid fumes have disappeared, 0·5 c.c. of a solution of 1 gramme of sodium hypophosphite in 100 c.c. of 1·12 HCl is run in. Evaporating a second time, a more or less distinct gray or black ring is produced, according to the amount of arsenic present; and the latter may be estimated by colorimetric comparison with a standard solution. To prepare such a standard, 0·264 gramme of As_2O_3 is boiled with nitric acid and diluted to 1 litre; 10 c.c. of the solution being again diluted to 1000 c.c. : 3 c.c. (equivalent to 0·006 milligramme of As) are employed in each test.

F. H. L.

The Use of Copper and Silver Thiocyanates in Gravimetric Analysis. R. G. van Name. (*Zeit. anorg. Chem.*, xxvi., 230.)—*Copper Thiocyanate.*—It was found that in the presence of free sulphuric or hydrochloric acid complete precipitation was insured by using an excess—more than twice the theoretical quantity—of ammonium thiocyanate in a fairly dilute solution, and letting the precipitate stand for at least twenty hours before filtering. The precipitate was filtered through asbestos placed in a perforated crucible, dried for two or three hours at 110°, and weighed as $Cu_2(CNS)_2$.

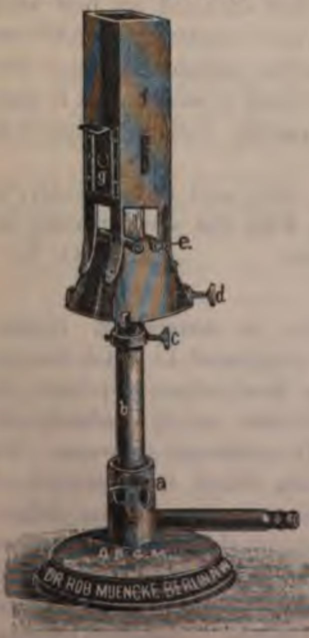
Silver Thiocyanate.—This compound may be used for the estimation of soluble thiocyanates. The solution is precipitated by adding an excess of silver nitrate, and the silver thiocyanate filtered, best after allowing it to stand for several hours, through asbestos dried for two or three hours at 110°, and weighed. The results were as satisfactory as those obtained by the use of Volhard's method.

A. G. L.

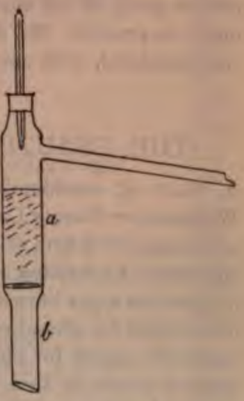
APPARATUS.

A Lamp for Monochromatic Light. F. W. Braun. (*Chem. Zeit.*, 1901, xxv., 69.).—In this burner the yellow light is derived from four rods of sodium laid in the metal coils marked *e*, shown in the annexed diagram. It is claimed that the lamp burns for a long time, is very cleanly, and is easy to manipulate.

F. H. L.



Fractionating Tube for Distillations under Diminished Pressure. W. Hirschel. (*Oesterr. Chem. Zeit.*, 1900, iii., 517.)—In this apparatus the wider tube *a* is about 90 mm. long and 20 mm. in diameter; it contains a platinum spiral of the same diameter as the glass, and in use is filled with glass balls 4 mm. in size. The narrower tube *b* is at least 15 mm. wide, and is fused to *a* after inserting the spiral. The device can be used with any flask; and at the end of a distillation the liquid retained among the balls is recovered by distilling a little ether through it. The ether expels the oil, etc., which falls back into the flask, and the superfluous ether can be removed by a current of air. The wire spiral is presumably claimed to permit the condensed distillate to return to the still during fractionation. F. H. L.



ANALYSTS' CERTIFICATES—WEIGHING THE SAMPLE.

(*Reprinted from the "Pharmaceutical Journal" of May* 18, 1901.)

In the Divisional Court on Thursday, May 9, the Lord Chief Justice and Mr. Justice Lawrance heard a case of some interest to chemists. This was an appeal—Sneath *v.* Taylor—against a conviction under the Sale of Food and Drugs Act, 1875, the facts of the case being that in November last the appellant, Alfred Petchell Sneath, an employé of Edward Allen and Sons, Limited (grocers), Sleaford (Lincolnshire), sold to the respondent, an Inspector under the Act, one pound of butter which, upon analysis, was certified to be adulterated with 15 per cent. of margarine, and was, therefore, not of the nature, substance and quality demanded by the purchaser.

The case for the appellant was that the certificate of the Public Analyst was invalid because it omitted the weight of the sample which was sent to him for analysis. It was contended that, as the whole evidence of adulteration was the certificate of the analyst, the person who produced a certificate with the omission of the weight should show that the article could not have been conveniently weighed—which, of course, was impossible in the present case.

For the respondent it was submitted that some meaning must be given to the word "conveniently" in Section 6 of the Act, and that it was a matter for the discretion of the analyst whether or not he weighed the sample.

The Lord Chief Justice said that where weight was material the law was plain that it should be filled in, but where it was not shown or suggested that the insertion of the weight had anything to do with the sufficiency of the certificate they could not decide, as a matter of law, that it must be filled in before the certificate could be acted upon. The analyst was allowed a certain amount of latitude, because it was provided that when the article could not be conveniently weighed the words might be erased or the blank left unfilled. In that case the magistrate would have no means of knowing, except by the omission of the weight, that the analyst thought it was inconvenient that it should be weighed. In the case under consideration, the article being butter, it could have been weighed, and, therefore, the analyst ought to have filled in the weight. If the accuracy of the analysis depended upon the weight being known or the various constituents being ascertained, or if the certificate was faulty in the sense that it did not give the requisite information, a different conclusion might be arrived at; but, it being a direction, he could not say that the simple omission of the weight, having regard to the discretion given to the analyst, was enough to enable the Court to hold that the objection raised ought to prevail. Mr. Justice Lawrance agreed with his Lordship's remarks, and the appeal was dismissed, with costs.

THE UNAUTHORIZED PUBLICATION OF ANALYSTS' REPORTS.

A POINT of considerable interest to analytical and consulting chemists was decided at the Westminster County Court on May 1, 1901, when an engineer and sanitary plumber brought an action against a firm of analytical and consulting chemists, claiming damages for "deteriorating the value of a written report by writing upon it." The plaintiff obtained from the defendants a report on a gas burner in the ordinary way. This report was subsequently returned to the defendants for alteration. The defendants' suspicions being aroused that the plaintiff intended to use the report for publication, they returned it to him with the words "Not for publication" written across its face. Plaintiff contended that this made the report valueless to him, and stated that he considered he had a right to use the report in any way he thought fit. The defendants defended the action on the ground that it attacked the principle that such reports were made entirely for the information of the person applying to them, and not for use by way of publication and advertisement generally.

It was contended on behalf of the defendants that they had a perfect right to object to the publication of their report, and that the plaintiff could acquire no right whatever to publish it unless a special and distinct agreement as to publication had been first entered into, and, further that, the defendants being the authors of the report, they had, at common law, a right and power to prevent any infringement of their right in a printed or written unpublished document. He quoted in support of this contention the cases of Lytton *v.* Devey, Hopkins *v.* Burghley, and others, and relied upon the law as laid down by Vice-Chancellor Malins in the first-named case as follows:

> "In my opinion the law upon the subject is plain, and has existed long before the case of Pope *v.* Curl was decided, and has existed ever since—namely, that the property in letters remains in the person to whom they are sent. The right to retain them remains in the person to whom the letters are sent, but the sender of the letters has still that kind of interest, if not property, in the letters which gives him a right to restrain any use being made of the communications which he has made in the letters so sent by him. I will not have it supposed that I entertain a moment's doubt about that being the settled law."

Evidence was called to show that it was the universal and recognised practice of the chemical profession to treat such reports as not for publication or advertisement without there being a special agreement permitting it.

In giving judgment, his Honour held that the defendants had established their contention that such a report was not for publication in the general sense, and that the plaintiff could not make use of it in the way he intended to. In the present case he saw a difficulty in that, the report having been once sent, it became the plaintiff's property, and that the defendants had exceeded their rights by writing across the report, after delivery, the words complained of, but that they would have been perfectly justified and within their rights in returning it with a covering letter stating that the report was not for publication, or in having written the words before the report originally left them. In the result his Honour held, however, that technically the defendants had exceeded their rights, but that there was no damage. He should therefore for the technical trespass award one farthing damages. This he afterwards altered to one shilling on its being pointed out that the County Court could not record a farthing. He, however, offered the defendants leave to appeal if so advised.

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THE ANALYST.

JULY, 1901.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE monthly meeting of the Society was held on Wednesday evening, June 5, in the Chemical Society's Rooms, Burlington House.

The President (Dr. J. Augustus Voelcker, M.A., B.Sc.) occupied the chair.

The minutes of the previous meeting were read and confirmed.

A certificate of proposal for election to membership in favour of Mr. F. M. Wharton, F.I.C., was read for the second time.

The President announced that it had been arranged that the summer meeting of the Society should be held at Cambridge on Friday, July 19.

The following papers were read: "Arsenic in Coal and Coke," by Alfred C. Chapman; "Public Analysts' Records," by J. F. Liverseege; and "The Use of Partially Sterilized Milk Cultures in judging the Purity of Water," by H. Droop Richmond.

ON THE MAUMENÉ TEST FOR OILS.

BY C. AINSWORTH MITCHELL, B.A., F.I.C.

(*Read at the Meeting, April* 3, 1901.)

IT was pointed out by Mr. Hehner and myself in a paper read before this Society in 1895 (ANALYST, xx., 146) that in certain cases there existed a rough proportion between the percentage of iodine absorbed by an oil and the Maumené figure, but that the factor applicable to different specimens of one kind of oil could not be applied to those of another kind, even when the determinations were made by the same person.

We hazarded the suggestion that, if the other thermal factors could be eliminated or made constant, the heat produced by the absorption of the sulphuric acid would be found to be directly proportional to the degree of unsaturation in the majority of cases.

In the ordinary methods of applying the test, there seems to be but little hope of attaining this end, for when concentrated sulphuric acid is added to an oil there is a strong local action, and it depends on the method and rate of stirring as to how soon a homogeneous mixture is obtained. This has a considerable influence on the rise of temperature.

Then, when highly unsaturated glycerides, like linseed and fish oils, are tested, it is practically impossible to get concordant results, owing to the higher temperatures obtained leading to further action on the part of the sulphuric acid, which it is not possible to keep within limits.

This was recognised by Maumené himself, who met the difficulty by mixing the oil with a definite proportion of olive oil of known thermal value, and subsequently making a correction for the addition.

Ellis (*Journ. Soc. Chem. Ind.*, 1886, 361) employed a mineral oil for the same purpose, but his method was further complicated by the fact that, the rise of temperature with the mineral oil and sulphuric acid alone in the blank determination being too slow, he had to mix the mineral oil with colza oil in order to obtain a more rapid reaction.

Both these methods suffer from the drawbacks of introducing active agents into the reaction, and of there being no possibility of different chemists obtaining diluting agents of exactly the same thermal value. Moreover, they only partially remedy the effects of overheating.

It occurred to me that carbon tetrachloride might be a suitable substance for the dilution, and that, if so, it would be possible for different observers to be sure of working under the same conditions.

On stirring 10 c.c. of carbon tetrachloride with 1 c.c. of sulphuric acid (93·8 per cent.), I found that the rise in temperature only amounted to 0·5° C. after one minute, and that this might be taken as a constant and neglected.

Method.—After some preliminary experiments I adopted the following method, so as to make the test comparable with the bromine thermal test devised by Mr. Hehner and myself: 2 grammes of the oil were weighed into a vacuum-jacketed tube,* 10 c.c. of carbon tetrachloride added, the temperature of the solution taken, and 2 c.c. of sulphuric acid introduced.

The liquid was continually stirred with a standard Jena thermometer graduated in tenths of a degree, and a note taken of the rise in temperature. Shortly before the end of the reaction the sulphonated product separated, and rose to the surface of the carbon tetrachloride. In this way duplicate results could be obtained agreeing within 0·2 of a degree. The time occupied by the reaction was about one minute, but to this point I shall return presently.

Fatty Acids.—I made the first experiments with the fatty acids themselves, so as to eliminate from the reaction the heat due to hydrolysis. In each case I determined the bromine thermal factor by our original method (ANALYST, xx., 146), and I give the iodine values calculated from the two thermal values in the subjoined table. The vacuum jacketed tube was standardized on almond oil (Hübl value, 96·5; bromine thermal value, 16·1 × 6 = 96·5) and lard (Hübl value, 64·6; bromine thermal value, 10·8 × 6 = 64·8).

---

* These can be obtained from C. E. Müller, High Holborn, W.C.

I.

Fatty Acids.

Oil.	Rise with H_2SO_4 (93·8 per cent.). ° C.	Calculated Iodine Value Rise × 7.	Bromine Thermal Rise. ° C.	Calculated Iodine Value Rise × 6.
1. Oleic acid I. (old)	11·8; 11·7	82·6	14·0	84·0
2. „ II. „	11·3; 11·4	79·8	14·3; 14·2	85·8
3. Almond	13·6; 13·8	96·6	16·1	96·6
4. Earthnut	12·5	87·5	14·2	85·2
5. Olive	11·6	81·2	14·1	84·6
6. Cotton	16·8	117·6	18·1	108·6
7. Maize I. (6 years old, rancid)	18·4	128·8	18·4	110·4
8. Maize II.	16·9	118·3	19·8	118·8
9. „ III. (1898)	17·2	120·4	20·0	120·0
10. Sesamé	16·0	112·0	18·1	108·6
11. Rape I.	13·7	95·9	16·5	90·0
12. Poppy (1895)	17·8	124·6	20·4	122·4
13. Linseed I. (4 years old) ...	24·8	173·6	28·2	169·2
14. „ II. (Calcutta) ...	24·7	173·9	29·4	176·4
15. „ II. (after 3 weeks)	27·6	193·2	27·9	167·4
16. „ III. (oxidized) ...	21·5	150·5	21·7	130·2
17. „ IV. (boiled 10 hours)	23·9	167·3	28·3	169·8
18. Tung I.	23·2	162·4	21·2	127·2
19. Castor	16·8	117·6	14·5	87·0
20. Cod-liver (old)	20·5	143·5	23·4	140·4
21. Whale (old)	14·0	98·0	15·3	91·8
22. Butter I.	8·1	56·7	5·8	34·8
23. „ II.	9·4; 9·1	65·8	6·0	36·0
24. Mutton-fat	9·6	67·2	8·6	51·6
25. Lard	11·5	80·5	10·9; 10·8	65 4

It will be seen that there is a close agreement between the two calculated iodine values with the majority of these oils. With the cottonseed oil (No. 6), the rise with sulphuric acid is too high; but I know nothing of the origin of that oil, and it is quite possible that it may have been exposed to the action of light and air for some time.

It appears from the results obtained with the maize oil acids (No. 7) that exposure of the oil to atmospheric influences affects the Maumené figure of the fatty acids in the opposite direction to the iodine value. In 1895 this same specimen of oil had an iodine value (Hübl) of 122, and calculated iodine value (from the bromine rise) of 118. The calculated figure has now fallen to 115, and the oil has become rancid; so that it is not surprising that the iodine value calculated from the Maumené figure should be too high. This inference receives support from the results of Ballantyne (*Journ. Soc. Chem. Ind.*, 1891, 29), who found the ordinary Maumené figure of different oils to be considerably increased after exposure to sunlight. The results obtained with the two other specimens of maize oil show a satisfactory agreement.

Coming next to the drying oils, the Maumené iodine value of No. 13 is too high;

but here, too, the bromine thermal value shows that atmospheric oxidation has taken place. In the case of No. 14, which was a sample of Calcutta oil from Mr. Williams, the agreement is very close. No. 15 shows the effect of exposing the fatty acids of No. 14 for three weeks to atmospheric influences. No. 16 was a sample which, having been left for four years in an open bottle, had become very viscous and was covered with a thick skin. No. 17 was a sample of oil which had been boiled for ten hours. Its calculated iodine values are in close agreement, as is also the case with the sample of poppy oil (No. 12).

The specimen of tung oil (No. 18) is the same one examined by Mr. Jenkins in 1898. The sulphuric acid rise is much too great in proportion to the bromine thermal value. The iodine value calculated from the rise of temperature with bromine (127·2) agrees well with that calculated by Mr. Jenkins three years ago (ANALYST, xxiii., 113).

It is not surprising that the Maumené iodine value of castor oil fatty acids should be too high, for sulphuric acid acts differently on ricinoleic acid and oleic acid, yielding an unsaturated product in the case of the former.

The values of cod-liver oil agree well, whilst in the case of whale oil, which was a sample of unknown age, the Maumené iodine value is too high.

The results obtained with the two specimens of butter fatty acids are interesting, and it requires further research to determine whether the higher Maumené figures were due to oxyacids, or to the action of the sulphuric acid on the lower insoluble fatty acids, or to alterations in the process of manufacture. I made one experiment with palmitic acid, and only obtained a rise of 0·2° C., after deducting 0·5° C. for the rise of temperature with carbon tetrachloride. I am inclined to attribute the want of agreement in the case of the mutton fatty acids (No. 24) and lard No. 25) to the effect of overheating during rendering, seeing that the Maumené rise is much more sensitive to the influence of external factors than the bromine thermal value.

Behaviour of Glycerides.—In the case of the oils themselves the agreement between the Maumené figure and the iodine value is, as a rule, not nearly so close as with the fatty acids, though there is still an evident proportion. The amount of heat due to the hydrolysis appears to vary in the different oils, and in some cases the strength of the sulphuric acid has a very great influence. It is possible, however, to obtain closely concordant results, and it may eventually be found possible to make a sharp distinction between the heat of hydrolysis and the heat of absorption. By the method which I have described it will also be possible for different chemists to obtain comparable results, and I would suggest that the figures obtained be calculated on the basis of an apparatus with a bromine thermal factor of 5, and that the exact strength of sulphuric acid be given.

Halogen Standard Figures.—The influence of using a different vacuum-jacketed tube and a different thermometer is shown in the following results, the acids used in each case containing 93·8 per cent. of anhydrous sulphuric acid:

II.

Oil.	Vacuum-tube I. Thermometer I. Bromine Factor = 6.		Tube II. Thermometer II. Bromine Factor = 5·52.	
	Temperature Rise with H_2SO_4 (93·8 per cent.).	Halogen Standard Figure.	Rise of Temperature.	Halogen Standard Figure.
	° C.		° C.	
Almond oil	12·7	15·24	13·6	14·96
Cotton oil	15·3	18·36	16·3	17·93
Oleic acid	11·7	14·04	12·5	13·75

Influence of the Duration of the Reaction.—As the rise in temperature with bromine only occupies about twenty-five seconds, whereas the rise with sulphuric acid lasts from forty-five seconds to one or even two minutes, with the weaker strengths of acid it seemed possible that a considerable error might thus be introduced if different apparatus were used and merely standardized on the bromine thermal figure.

I therefore made a number of experiments with different oils to determine the probable loss of heat.

A correction, which is rough from the physicist's point of view, but which is sufficient for ordinary purposes, can be obtained by noting the fall of temperature from the highest point attained at intervals of five minutes, and dividing the total loss of temperature by the number of minutes before the mercury ceases to fall. It becomes apparently constant at 2° or 3° above the temperature of the room, which points to the establishment of an equilibrium between any substitution which occurs and the loss of heat. The loss thus determined amounted to approximately 0·2° C. per minute with different tubes, strengths of acid, and oils. If the rise does not occupy more than a minute, this correction lies within the limit of experimental error, and may be neglected.

It is advisable, however, to use acid of more than 96 per cent. strength in order to reduce this time error as far as possible.

Influence of Different Strengths of Acid.—Archbutt (*Journ. Soc. Chem. Ind.*, 1886, 304) showed that the concentration of the sulphuric acid had a considerable influence on the rise of temperature in the ordinary method, and this is also the case with the method I have described, as is seen by the following results:

III.

RISE OF TEMPERATURE WITH ACID OF DIFFERENT STRENGTHS.

Oil.	93·8 per cent.	95·1 per cent.	96·3 per cent.	97·8 per cent.
	° C.	° C.	° C.	° C.
Oleic acid	11·8	13·25	14·6	15·7
Almond oil	12·7	14·4	16·2	18·8
Cotton oil	15·0	18·1	20·0	22·9
Linseed oil	19·6	27·5	28·1	31·2

It will be observed that the rise with 93·8 per cent. acid was disproportionately low in the case of the linseed oil.

IV.

HALOGEN STANDARD MAUMENÉ FIGURES (CALCULATED ON BROMINE THERMAL FACTOR = 5).

Oil.	H_2SO_4 (93·8 per cent.).	Calculated I Value Rise × 6·35.	H_2SO_4 (97·8 per cent.).	Calculated I Value Rise × 4·3.	Bromine Thermal Value.	Calculated I Value Rise × 6.
	° C.		° C.		° C.	
1. Almond	15·2	96·5	22·4	96·3	16·1	96·6
2. Earthnut	14·0	88·9	20·5	88·1	14·3	85·8
3. Olive	12·7	80·6	18·4	79·1	13·8	82·8
4. Cotton	18·3	116·2	26·6	114·4	17·5	105·0
5. Maize I. (rancid) ...	24·5	155·5	34·4	147·9	19·2	115·2
6. ,, II. (old)	19·4	123·2	29·7	127·7	19·5	117·0
7. ,, III. (1898) ...	19·2	121·9	28·6	122·9	19·4	116·4
8. Sesamé	18·0	114·3	25·3	108·8	17·8	106·8
9. Mustard husk	18·2	115·6	26·6	114·3	18·2	109·2
10. Rape I.	17·6	111·7	23·9	103·7	16·7	100·2
11. ,, II.	17·0	107·9	23·2	99·7	15·0	90·0
12. ,, III. (Japan) ...	15·0	95·2	22·0	94·6	16·7	100·2
13. Poppy (old)	25·5	161·9	38·0	163·4	21·0	126·0
14. Linseed I. (4 years old)	24·1	153·0	37·2	159·9	27·3	163·8
15. ,, II. (Calcutta) ...	23·4	148·5	37·3	160·3	28·2	169·2
16. ,, III. (oxidized) ...	26·0	165·1	39·9	171·5	22·2	133·2
17. ,, IV. (Baltic) ...	22·3	141·6	37·2	159·9	30·2	181·2
18. ,, V. (1898) ...	24·5	155·5	37·8	162·5	29·3	175·8
19. ,, VI. boiled 10 hrs.	21·6	137·0	35·0	150·5	27·6	165·6
20. ,, ,, ,, 15 ,,	21·7	167·7	22·3	95·9	27·4	164·4
21. ,, ,, ,, 20 ,,	13·9	88·2	24·4	104·9	19·5	117·0
22. Tung I.	12·6	80·0	33·9	145·8	21·2	127·2
23. ,, II.	—	—	26·2	112·6	23·7	142·2
24. Castor	20·8; 20·7	132·0	27·6	118·6	15·0	90·0
25. Cod-liver (old) ...	—	—	38·5	165·5	24·5	147·0
26. Whale	12·8	81·0	18·6	79·9	15·8	94·8
27. Butter I.	11·0	69·8	—	—	6·2	37·2
28. ,, II.	10·6	67·2	—	—	5·4	32·4
29. Mutton-fat	9·2	58·4	—	—	8·3	49·8
30. Lard	11·0	69·8	16·5	70·9	10·8	64·8

If we compare these results, we see that the agreement between the different calculated iodine values is fairly satisfactory in the case of almond, earthnut, olive, maize (III.), sesamé, mustard husk, rape (I.) and Japanese rape oils.

The Maumené values of the cottonseed oil are too high, which was also the case with the fatty acids. All the specimens of rape oil were not less than three years old, whilst the poppy oil was more than six years old. This is probably the cause of the Maumené figures being too high.

The figures given by the linseed oils were invariably too low, but it is interesting that the results obtained with the 98 per cent. acid approximated more nearly to the truth. It is not improbable that complete proportion between the bromine and

Maumené figures of unoxidized oils might be obtained by the use of monohydrated sulphuric acid.

These results also confirm those given in Table III., and show that the oils are affected to a much greater extent than their fatty acids by the strength of the sulphuric acid. In the case of the boiled oils the Maumené results cannot be altogether trusted. On the addition of the acid, the entire amount of oil formed a clot round the thermometer, and it required very vigorous stirring to break up the mass, whilst even then the mercury rose but slowly.

An analogous behaviour was observed in the case of the two samples of tung oil, of which No. 1 was Mr. Jenkins's sample, and No. 2 was given me by Mr. Rowland Williams. This curious property of tung oil has already been noticed by Mr. Jenkins (ANALYST, 1898, xxiii., 114). The second sample was that examined by Mr. Rowland Williams (*Journ. Soc. Chem. Ind.*, 1900, 301).

The samples of cod-liver oil and whale oil were both old, and it is not surprising that the Maumené figures are too high. The results obtained with the samples of castor oil, butter, mutton-fat and lard show an excess over the bromine thermal figures, as was also noticed with their fatty acids. In certain instances the rise of temperature with the lower strength of acid was proportionately greater than with the higher strength. I am unable to offer a satisfactory explanation for this, except that the greater proportion of water in the lower strength of acid may have an influence on some secondary hydrolysis of the sulphonated product.

To summarize the foregoing results:

1. The Maumené figure determined by the method described above is in direct proportion to the bromine thermal value of the fatty acids of most unoxidized oils.
2. Castor oil, and apparently butter and animal fats rendered at a high temperature, form exceptions to this rule.
3. This relationship also applies to the glycerides, though these appear to be more influenced by atmospheric oxidation than their fatty acids.
4. The strength of the sulphuric acid has a great influence on the proportional results obtained with the glycerides, and even with 98 per cent. sulphuric acid the Maumené figures of unoxidized linseed oils are too low.
5. This modification of the Maumené test may be found useful in determining the degree of oxidation of fats and oils, inasmuch as the figure becomes greater with the decrease in the iodine value.

In conclusion, I have to express my best thanks to Mr. Rowland Williams for samples of linseed and boiled linseed oil, maize oil (II. and III.), and tung oil (II.), to Mr. Jenkins for the sample of tung oil (I.), and to Mr. Skertchly for checking the standardization of my tubes.

DISCUSSION.

Mr. JENKINS said that a weak point of the Maumené test hitherto had been that, in the case of oils like linseed and fish oils, some method of dilution was necessary to prevent foaming over, and the dilution of these oils led to results which were not easily comparable with those given by the non-diluted oils. This weak point Mr. Mitchell had met by the dilution of all oils alike. A further advantage of the method now described was apparent when the figures were compared with some results given in a paper which he (Mr. Jenkins) had read in 1897 before the Society of Chemical Industry, when about a dozen oils were compared with regard to their Hübl values, bromine temperature reactions (calculated to iodine values) and specific temperature reactions, the latter being the results of the Maumené test compared with the rise of temperature given by water under the same conditions, the latter being taken as 100. It will be seen that in the case of such non-drying oils as neat's-foot oil and olive oil the figure for specific temperature reaction was only about 1·2 times that of the Hübl figure; but as one approached the drying oils the increase of the Hübl figure was accompanied by a much greater rate of increase in the Maumené figure, the ratio then becoming more nearly 2. One of the interesting points of the present paper was that, with this new method of applying the test, such differences of ratio largely disappeared. Possibly in the earlier methods the actual rise of temperature with the drying oils was so great as to induce secondary reactions evolving more heat, whereas in Mr. Mitchell's experiments the oils were so largely diluted with the carbon tetrachloride as to prevent this undesirable secondary rise of temperature. The author's results with tung oil were especially interesting. With the tung oil (I.), when 93·8 per cent. acid was used, the rise of temperature was 12·6°, whereas when the acid was 97·8 per cent. the rise of temperature was 33·9°. It was also peculiar, inasmuch as the iodine value calculated from the bromine thermal value was considerably lower than the iodine value calculated from the Maumené figure. On the other hand, in the case of tung oil (II.), the calculated iodine value from the sulphuric acid test was much lower than that from the bromine thermal value.

Mr. Hehner said that, it being established that bromine was simply added on, and that the heat of bromination was simply the heat of combination of certainly two, and sometimes four, or even six, atoms of bromine, it seemed to him that the process might be regarded as, generally speaking, a uniform additive process; and seeing that in most cases the rise of temperature with sulphuric acid agreed fairly well with the bromine thermal value, it seemed as though the ordinary action of sulphuric acid upon an oily solution was simply a sulphonation process. Coming to the oxidized oils, there was something further to be considered. The oil contained an additional quantity of oxygen, which the sulphuric acid displaced, itself adding on and forming a sulphonated oil, while at the same time water was formed, which increased the rise of temperature; so that in the case of an oxidized oil the rise of temperature produced by the action of sulphuric acid was considerably increased. Similarly, there was a largely increased rise of temperature in the case of many oils which were of the nature of hydroxy compounds. The constitution of tung oil was peculiar. It had no oleic acid, or, if any, very little; but, on the other hand, it contained another unsaturated fatty acid, which crystallized in a magnificent manner, and which, he thought, probably contained three atoms of oxygen. It was quite certain that these Japanese wood soils differed considerably from one another. After all, when it was recognised that, by reason of slight oxidation or of the age of the oil, the factors were in some cases deeply influenced by the strength of the sulphuric acid employed, the analyst would naturally consider that probably he would not get much advantage from a reaction of this kind in dealing with an oil the constitution of which might be quite unknown to him. The scientific chemist, however, would not contemplate all these reactions merely with regard to the assistance they might afford him in his business, but would probably look deeper into the question, and he (Mr. Hehner) personally felt indebted to the author for the addition to knowledge that resulted from this work.

Mr. MITCHELL said that in diluting with olive or any other oil one could never be quite sure about the oil used. Ellis had pointed out some fifteen years ago that if the rise of temperature was above 60° C. it was perfectly impossible to get concordant results, and his (Ellis's) attempts had all been directed towards keeping the rise of temperature below 60°. It might be mentioned that the use of the particular form of tube now described did not seem to be absolutely essential. He had made experiments with an ordinary boiling test-tube packed inside a beaker with asbestos wool, the bromine thermal factor of this being determined; and provided the loss of heat was not too great, or was taken into account, this arrangement answered just as well as the vacuum-jacketed tube. In the case of tung oil some difficulty was experienced owing to the clot which it formed with sulphuric acid. It was necessary to stir vigorously for four or five minutes, and even then one could not be quite sure that the full rise of temperature was obtained.

ARSENIC ESTIMATIONS RELATING TO MALT-KILNS.

BY T. FAIRLEY, F.I.C.

(Read at the Meeting, April 3, 1901.)

As one result of the close attention and supervision which has lately been given to materials, etc., relating to the malting and brewing industries, the occasional presence of arsenic in malt has been established. This arsenic has been traced by a number of persons (Thomson and others) to the fuel used in kilning the malt, and in consequence, in many cases, fuel practically free from arsenic has been sought for.

Having occasion to inspect same malt-kilns, it struck me that where an impure fuel had been used for a length of time the change to an arsenic-free fuel might not give the security from contamination of the malt which would be expected.

When one considers how readily arsenical vapour is condensed on any surfaces colder than itself, the walls and floors, etc., of malting-kilns might be expected to show arsenic in the dust deposited on them where an arseniferous coke has been used. I have been greatly surprised at the comparatively large proportions of arsenic which I have found under these circumstances.

The tiles forming the floor of the malting-kiln and the walls above and below the malting-floor act as condensing surfaces for the arsenical vapours given off from impure fuel, and the arsenic appears to accumulate on them, so that the effect of the minute proportion of arsenic in the fuel may be greatly magnified.

At first this condensing action, so far as it protects the malt from contamination, may be beneficial. After a time, given walls and tiles already charged with arsenic, it is obviously futile to change from an impure to a pure fuel so long as the tiles and surfaces through or over which the products of combustion must pass, on their way to the malt, are heavily charged with arsenic.

Some of the older tiles are unglazed and porous. In these the arsenical vapour has penetrated into the mass of the tile. Thus, in tiles weighing about 13 pounds, and measuring about 1 foot square, I have found from 109 to 36 grains of arsenic trioxide. It is evident that such tiles should be at once removed from the floor of the kiln.

Glazed tiles differ in the perfection of the glaze, according to age and manufacture. In these I have found from 25 grains of arsenic trioxide down to none—that is, when the dust adherent chiefly to the lower surface of the tiles had first been removed. In my opinion, tiles in which the arsenic has penetrated into the mass of the tile should in every case be removed.

In all cases where impure fuels have been used there cannot be too much care taken to remove all dust from tiles, floors, walls, crevices, etc., and only when this precaution is taken, and new tiles put in the floors where necessary, can the malt be protected from contamination by the change to a pure coke or fuel.

The proportion of arsenic in some of the dust adherent to the tiles is extraordinary, ranging from 1 down to 0·25 per cent. of the weight of the dust. The amount in the dust adherent to the walls is much less, from 0·06 down to 0·0025 per cent.

As the malt is only exposed for a limited time to the kilning process, the arsenic, where present in malt, is generally external, and can be removed by careful cleaning and brushing, etc., of the malt. Thus, the dust from the malt-screening machines contains arsenic frequently where none can be found in the cleaned malt. At the same time, it is sufficiently obvious that it is not right to trust to any merely mechanical process being carefully carried out perfectly, where such a dangerous substance as arsenic in a food, or article used in the preparation of food or drink, is concerned. The malt-screening process, however good in itself, was never intended to deal with such a serious problem as the removal of arsenic from the malt.

METHODS OF ANALYSIS.

The Tiles.—These were broken up and roughly pounded, then a smaller portion finely pounded and 2 to 5 grammes treated with 20 to 50 c.c. 25 per cent. sulphuric acid. In the older tiles there is calcium carbonate, and when the effervescence due to this has ceased the material is heated one to two hours on the water-bath.

Afterwards the volume of the liquid with the powder in suspension is made up to 50 or 100 c.c., and a measured portion of this put into the Marsh's apparatus. In this about 2 inches of the exit tube was heated to redness for thirty minutes. By comparison with a standard set of mirrors from known amounts of arsenic an approximate estimation of the arsenic in the tile is quickly made.

For the gravimetric estimations 10 to 20 grammes of the finely-pounded tile was treated and distilled with pure hydrochloric acid with the addition of ferrous chloride, until no more arsenic chloride came over. The arsenic precipitated as sulphide was collected, dried, and weighed. It was completely soluble in dilute ammonia.

The Dusts.—One to 5 grammes were treated with from 10 to 50 c.c. of strong sulphuric acid and about 20 c.c. nitric acid 1·42 specific gravity. When the organic matter was oxidized and the nitric acid boiled off, the liquid was allowed to cool, diluted and boiled to drive off oxides of nitrogen, and finally cooled and made up to 50 to 100 c.c. A fraction of this liquid was taken for the Marsh's test to bring the amount of arsenic within the range of the mirrors.

For the gravimetric tests 5 grammes of each dust was distilled with HCl as before, and the estimation finished as with the tiles.

I have found it difficult to obtain pure sulphuric acid capable of standing a prolonged Marsh test. For those who have similar difficulty the following method of purification may be recommended.

Many years ago I found that sulphuric acid distils quietly, like alcohol or water, in presence of alkaline bisulphates, and Maxwell Lyte and others showed that arsenic in the form of arsenic acid is not volatile with sulphuric acid.

A convenient-sized retort is about half filled with commercial "pure" sulphuric acid, to which has been added 25 to 30 per cent. of sodium or potassium bisulphate, or proportionately less sulphate. To this is added potassium permanganate or bichromate in small quantity to secure complete oxidation of any arsenic that may be present. Heat is applied by means of a gauze, rose, or ring burner, and the whole arrangement carefully screened from draughts, which, cracking the hot glass, are almost the only source of danger. About two-thirds of the acid may be distilled over.

If potassium bichromate be used, then gradually an anhydrous chromium sulphate is formed, which, settling at the bottom as a heavy powder, causes bumping, necessitating the use of a ring burner.

If this method be used to purify *ordinary* commercial acid, ammonium sulphate may be added in place of other alkaline sulphate, and acts like them. It also reacts with and destroys any oxides of nitrogen that may be present, after which the oxidizing agent may be added.

MALT-KILNS.

Percentages of Arsenic Trioxide in Tiles and Dusts.

A. Floor tiles, unglazed: 0·12 ; 0·064 ; 0·04.
B. ,, ,, glazed and cleaned: 0·03 ; 0·02 ; 0·02 ; 0·01 ; 0·008 ; none.
C. ,, ,, dust from under surface: 1·0 ; 0·8 ; 0·75 ; 0·7 ; 0·3 ; 0·2.
D. Malting-room, dust from walls above floor: 0·06 ; 0·04 ; 0·035 ; 0·03 ; 0·02.
E. Chamber or space below malting-room, dust from walls, etc. (often chiefly culms): 0·05; 0·035 ; 0·03 ; 0·025 ; 0·012 ; 0·008 ; 0·007 ; 0·004 ; 0·0025. The dust giving 0·004 per cent. As_4O_6 was sifted to separate culms, and the fine dust then gave 0·07.
F. Malt-screening machines, dust containing much organic matter: 0·01 ; 0·004 ; 0·0016.

Some of the above estimations were repeated gravimetrically, with the results

			Gravimetric Test.		Mirror Test.
			As_4S_6 Grammes.	As_4O_6 %	As_4O_6%
A. Tiles	...	... 10 grammes gave	0·0128	0·10	0·12
,,	...	... 20 ,, ,,	0·0179	0·072	0·064
C. Dusts	...	... 5 ,, ,,	0·0659	1·06	1·00
,,	...	... 5 ,, ,,	0·0465	0·74	0·80
,,	...	... 5 ,, ,,	0·0464	0·74	0·75
,,	...	... 5 ,, ,,	0·0410	0·66	0·70

I hope these results may prove useful to maltsters and others interested in helping them to avoid a possible source of danger—even where a pure fuel is now adopted.

Mr. A. B. Shepherd, B.Sc., has assisted me in the analytical work in this paper, and I have pleasure in stating that my best thanks are given to him.

DISCUSSION.

Mr. BAKER said that he had recently examined several samples of dust collected from malt-houses of the more modern type, in which the heated air was diffused by means of a "baffle plate" fixed above the fire and underneath the malting-floor. The fuel used would be coke, or a mixture of anthracite and coke, or if coal only were used the kilning operation would in some cases be concluded with a small quantity of foundry coke to "finish off the goods." The average quantity of arsenic present in the dust from different parts of the kiln was about $\frac{1}{5}$ of a grain per pound. The dust which collected underneath the baffle plate seemed to be very rich indeed in arsenic, containing from $1\frac{1}{2}$ to 2 grains per pound. On the top of the baffle plate a large quantity of malt coombs and dust collected, and these were comparatively poor in arsenic. When the malt came off the floor it was subjected to a process of screening. In the case of malt of very good quality containing in the finished state not more than $\frac{1}{500}$ grain of arsenic per pound, the coombs from the last screen would contain as much as $\frac{1}{33}$ grain of arsenic per pound. With regard to the question of whether brushing would remove arsenic from malt, he thought that when a large quantity of arsenic was present possibly a portion of it might be removed in this way, but that when the quantity of arsenic was small—say $\frac{1}{300}$ or $\frac{1}{400}$ grain per pound—it made absolutely no difference whether the malt was brushed or simply cleaned in the ordinary way.

Mr. FAIRLEY said that the older kilns consisted, as it were, of one room on the top of another, the upper room having the malting-floor, while the lower, which had very thick walls, contained the fireplaces, the products of combustion passing upwards through tunnels in the side walls. The sample of dust that contained 1 per cent. of arsenic was from one of the modern arrangements, while that which contained 0·8 per cent. was from an old-fashioned kiln.

THE GUTZEIT TEST FOR ARSENIC.

By F. C. J. Bird.

(*Read at the Meeting, April* 3, 1901.)

The Gutzeit method of detecting arsenic, like that of Marsh, depends primarily on the formation of arsine by the action of nascent hydrogen generated from zinc and hydrochloric or sulphuric acid in contact with the arsenic-containing substance. Originally the arsine was allowed to act on either nitrate of silver or mercuric chloride paper; but so many objections apply to the use of the former, that latterly the term "Gutzeit's test" has come to be associated with the employment of mercuric paper only, on which arseniuretted hydrogen produces a yellow stain. The convenience and simplicity of this test would doubtless cause it to be far more generally employed were it not for the occasionally uncertain and misleading nature of its indications.

The chief objections to the method, as ordinarily carried out, may be summarized as follows:

1. It is not absolutely negative. Traces of arsenic associated with such substances as certain coal-tar colours, compounds reducible by nascent hydrogen, soaps, etc., or existing as arsenates in very small proportion, may escape detection.

2. Unless the state of oxidation of the arsenical compound is known, it fails as an approximately quantitative method; for any attempt to deduce the amount of arsenic present from the depth of the stain is likely to result in error, on account of the fact that arsenates yield a much paler stain in a given time than arsenites. The intensity of the stain is also influenced by the degree of humidity of the mercuric chloride paper. Water, especially when warm, dissolves the stain completely, leaving the paper white; dampness and extreme dryness of the mercuric chloride paper are alike prejudicial to the production of a stain of correct intensity.

3. Stains which either resemble or may mask or deepen a true arsenical stain are produced by the hydrides of phosphorus, sulphur, and antimony. Sulphuretted hydrogen (and also selenium hydride) can be absorbed by lead acetate solution; but phosphoretted hydrogen, always a possible impurity, passes through the liquid, and tints the mercuric chloride paper full yellow. Acid cuprous chloride solution was tried as an absorbing agent for this interfering compound, but proved unsatisfactory. Antimoniuretted hydrogen gives a yellowish-brown to full brown and black coloration, which gradually fades on exposure to air. When dealing with colourless inorganic salts, such as phosphate of soda, etc., oxidation by iodine, bromine, or permanganate is an effective safeguard against both phosphine and hydric sulphide; but with some organic substances either the halogen is absorbed, or the permanganate reduced almost indefinitely; and as with an excess of either oxidizing agent the presence of arsenic may be completely masked, preliminary oxidation has but a limited application.

4. No simple method has hitherto been described either of distinguishing between arsenical and interfering stains, or of demonstrating the real nature of a supposed arsenical stain.

From the foregoing considerations it is evident that the Gutzeit test in its presen form is at best but a preliminary test, not admitting of even approximately accurate quantitative deductions (excepting in a few special instances), and occasionally liable to mislead on account of the uncertainty of the absence of phosphoretted compounds (especially in coloured substances), or of traces of sulphuretted hydrogen unabsorbed by the lead acetate during a sudden rush of gas. On the other hand, if the difficulties mentioned could be satisfactorily overcome, the test itself offers several obvious advantages.

If uniform results are to be always obtained with tests like those of Marsh and Gutzeit, it is essential that the evolution of arsine take place under fixed and constant conditions. The rate of formation of arsine appears to be regulated, firstly, by the concentration of the arsenical solution, and, secondly, by the energy of the reaction between the zinc and acid, this latter being dependent on the strength of the diluted acid, and especially on the temperature of the liquid. It can be easily demonstrated that in similar solutions 100 square centimetres of mercuric chloride paper are coloured to a full yellow by 1 milligramme As_4O_6 by the Gutzeit reaction in a few minutes, whilst the same effect is not produced on 1 square centimetre by $\frac{1}{100}$ milligramme As_4O_6 in less than ten minutes or more, all other conditions being precisely equal. So also in the Marsh-Berzelius test, when the time limit is half an hour, it is generally noticed that the greater part of the arsenic is deposited during the second fifteen minutes, when the liquid in the generating flask has become warm and the chemical action energetic. Uniformity of conditions would therefore appear to be best insured by: (*a*) The complete solution of a given weight of zinc in a given time; (*b*) the maintenance of a constant temperature in the generating flask; and (*c*) the limitation of the volume of the solution in the generating flask to a given quantity. I have accordingly taken 4 grammes of zinc, completely dissolved in fifteen minutes at the boiling temperature of a liquid measuring not more than 100 c.c., as the basis of a satisfactory method of conducting the Gutzeit test, and have found the same principle of reaction at a boiling temperature to apply equally well to the Marsh-Berzelius method.

The apparatus employed for this modification consists of a flat-bottomed 150 c.c. flask, furnished with a small upright reflux condenser and a 10-inch thistle funnel, above which is supported a tap-separator for the regular supply of acid to the flask. The exit tube of the condenser is curved over, and passes into a small wash-bottle containing 10 per cent. lead acetate solution, whence a suitable tube carries the gas to the mercuric chloride paper disc. Four grammes of arsenic-free zinc in very small fragments are placed in the flask, together with the suspected substance, dissolved in 30 c.c. water. The solution is heated to the boiling-point on a hot-plate, and kept at that temperature by a small flame underneath; 15 c.c. of pure hydrochloric are then gradually run in by the tap-funnel, a steady evolution of gas being maintained by adjusting the stopcock, so that the acid is supplied in a regular succession of drops. The rate of flow should be such that the whole of the acid is delivered in ten minutes; the contents of the flask are then boiled for five minutes longer, when the zinc will have disappeared and the test may be considered completed. The quantity of acid used is in all cases half the volume of the liquid in the flask. Seventy c.c. of beer, for example, require 35 c.c. of acid, all of which should be run in during the ten minutes. Whenever possible, it is convenient to keep the volume of the solution within 30 c.c.; but with original weakly arsenical liquids, as beer, the limit can be extended to 70 c.c. without disadvantage.

A similar arrangement with the zinc, etc., proportionately increased if necessary, suffices for the adaptation of the same principle to the Marsh-Berzelius method. Lead acetate solution is more effective in keeping back sulphuretted hydrogen than the roll of dry lead paper often recommended in analytical treatises. Traces, even, of sulphuretted hydrogen have a decided influence on the appearance of the Marsh-Berzelius mirror, and to the presence of small quantities of this gas in the mixture of hydrogen and arsine, which have escaped fixation by the lead paper roll, I am inclined to attribute the greater sensitiveness of hydrochloric acid as compared with sulphuric acid when used in the Marsh-Berzelius apparatus. The purest analytical sulphuric acid diluted and allowed to act on pure zinc almost invariably yields a gas which quickly blackens moist lead paper; hydrochloric acid similarly treated usually gives but a faint reaction. A comparison of the effect of the two acids with known quantities of arsenic, the sulphuretted hydrogen having been completely removed by lead acetate *solution*, has shown no perceptible difference in the resulting mirrors. Hydrochloric acid is, however, the more convenient for the reasons given by Mr. Otto Hehner some weeks ago when describing his admirable method of applying the Marsh-Berzelius process to the quantitative determination of minute traces of arsenic.

The powerful reducing action of nascent hydrogen evolved at the boiling temperature of the liquid in this modified Gutzeit test renders possible the detection of traces of arsenic in certain combinations which in the cold are liable to be missed. Under these conditions the stain from an arsenate is practically identical with that from its equivalent of arsenite; even $\frac{1}{100}$ milligramme as arsenate makes its presence evident within two or three minutes. The time requisite for an experiment is reduced to fifteen minutes, and neither sulphurous acid nor substances themselves reducible by nascent hydrogen interfere with the formation of arsine. Even dry precipitated sulphide of arsenic ($\frac{1}{50}$ milligramme) readily yields its arsenic at the boiling temperature if the solution contain water and acid only; much glucose, however, interferes with or entirely prevents reaction.

Beer and similar liquids require no preliminary treatment. Frothing, often so troublesome with beer samples, does not occur at a boiling temperature, the volatilization and condensation of the alcohol on the surface of the liquid effectually breaking up the froth as it forms; 2·5 to 5·0 c.c. of alcohol added to the liquid in the generating flask acts in a similar manner with substances containing dextrin, etc., which from the same cause are often equally difficult to deal with.

The limit of sensitiveness of the Gutzeit test has been variously stated. One-hundredth milligramme As_4O_6 (or considerably less of arsenic acid) is a figure which has been given quite recently, whilst in the ANALYST for 1891 Curtmann states that $\frac{1}{1000}$ milligramme in 1 c.c. produces a yellow stain in thirty minutes, and that $\frac{1}{10000}$ milligramme gives a barely perceptible stain after one hour. My own experience points to $\frac{1}{20}$ milligramme of an arsenate and $\frac{1}{50}$ milligramme As_4O_6 as about the smallest amounts capable of producing a distinctly recognisable stain with a time limit of half to one hour in the ordinary way of applying the test. Just, however, as the sensitiveness of the Marsh-Berzelius method can be enormously increased by contracting the diameter of the heated tube, so also by decreasing the area of the mercuric chloride disc does the Gutzeit test gain in delicacy. The limit of sensitiveness of the modified method with a 5 millimetre disc appears to be $\frac{1}{100}$ milligramme As_4O_6; whether existing as arsenate, sulphide in an aqueous liquid, or mixed with sulphurous acid, soap, reducible coal tar compounds, etc., is immaterial. This quantity of arsenic, reacting at a boiling temperature, stains a 5-millimetre mercuric chloride paper disc to full intensity in about ten minutes, and the stain is capable of being identified without difficulty by two reactions to be subsequently described. Stains are obtainable with much smaller amounts of arsenic (perhaps $\frac{1}{1000}$ milligramme), but such stains do not admit of satisfactory confirmation.

A small piece of apparatus useful for the concentration of the arsenical stain consists of a short piece of wide glass tube contracted at one end to a diameter of 5 millimetres, the contracted extremity being ground flat. A disc of mercuric paper (filter-paper saturated with 5 per cent. mercuric chloride solution, dried, folded, and cut into circles with a suitable cork borer) is attached to the ground extremity of the tube previously smeared with thick mucilage. A second similar tube bearing a paper disc is placed over the first, the joint being either a ground one or extemporized with rubber tubing, and the whole is surmounted by a third tube, the extremity of which is drawn out and curved over in order to prevent the gas from escaping too freely. The first or lower tube, drawn out below to a smaller diameter, communicates with the lead acetate wash-bottle or bulb previously mentioned. By this arrangement the hydrogen mixed with arsine passes through the pores of the mercuric paper under some pressure and thorough deposition of the arsenic is ensured; the pressure existing in the apparatus is usually represented by 6 or 7 inches of liquid in the funnel tube.

The absence of any means of demonstrating the true nature of the Gutzeit stain has long been recognised as the most fatal objection to the test. In order to remove this defect if possible, I have investigated the action of various reagents, and have found that treatment of the stain with boiling hydrochloric acid affords a ready means of identification, and that its arsenical nature can be subsequently confirmed by a special application of Bettendorff's stannous chloride reaction.

On submitting the stains produced by SH_2, PH_3, and AsH_3 to the action of water alone, the following effects are observed: The sulphuretted stain is hardly affected by cold, but is almost entirely dissolved by hot water. The stain due to phosphoretted hydrogen becomes of a paler but brighter lemon yellow with either hot or cold water, whilst water even but slightly warm washes out both the mercury and arsenic compounds from the arsenical stain, leaving the paper colourless. The subjoined table exhibits the marked distinction drawn by hot strong hydrochloric acid between stains on mercuric paper due to sulphuretted hydrogen, phosphoretted hydrogen, antimoniuretted hydrogen and arseniuretted hydrogen:

	Appearance of Stain.	Strong Hydrochloric Acid.	
		Cold.	Boiling.
SH_2.	Yellow to dull brown.	Stain dissolves slowly.	Stain dissolves immediately, leaving paper colourless.
PH_3.	Yellow.	Bright lemon-yellow.	Somewhat intensified; bright lemon-yellow.
SbH_3.	Brown-yellow to blackish-brown; fades on exposure to air.	Stain dissolves slowly.	Dissolves immediately; paper retains a gray tint.
AsH_3.	Yellow to orange and orange-brown.	Stain intensified; full lemon-yellow to orange.	Stain further intensified; brick-red to deep red-brown.

In the practical application of the above reactions, the stained discs of mercuric chloride paper are detached from the tubes by touching the edges of the discs with a moistened glass rod. They are placed in a watch-glass, about 1 c.c. of pure HCl (free from Cl) added, and the whole heated over a flame protected by an asbestos sheet until the acid boils. The liquid is then poured off and rejected, and the operation repeated. With $\frac{1}{100}$ millgramme of arsenic, the first disc will now be brick-red, and the second a faint orange. The treatment with HCl removes the salt of mercury, which would otherwise render the stannous chloride confirmation less distinct. The discs are now warmed with $\frac{1}{2}$ c.c. HCl, and the arsenical compound dissolved by the addition of one or more drops of bromine-hydrochloric acid, avoiding any large excess. The pale yellow liquid is poured off into a small test-tube 3 inches × $\frac{1}{4}$ inch, any residual acid displaced with one or two more drops of HCl, an equal volume of 30 per cent. stannous chloride solution in HCl added, and the contents of the test-tube warmed, when, if $\frac{1}{100}$ milligramme arsenic be present, the characteristic pink-brown coloration of Bettendorff's reaction makes its appearance almost immediately. With $\frac{1}{100}$ milligramme these reactions are very definite, and the depth of tint for that quantity, both with boiling hydrochloric acid and subsequently with stannous chloride, becomes easily recognisable. Phosphorus, sulphur, and antimony stains, when similarly treated, give negative results. The bromine-hydrochloric acid is strong hydrochloric acid containing sufficient bromine to impart a deep yellow colour. The stannous chloride reagent is prepared by dissolving 30 grammes of crystallized stannous chloride in 150 c.c. of pure hydrochloric acid and boiling down with a few fragments of metallic tin to 100 c.c. It should remain perfectly colourless when heated with an equal volume of hydrochloric acid and allowed to stand for some time.

The brick-red to deep red-brown colour produced by boiling HCl is very characteristic. Even in a mixed stain from $\frac{1}{100}$ milligramme As_4O_6 with hypophosphite and much sulphite (the SH_2 being purposely unabsorbed), the reddish tint due to arsenic is distinctly perceptible, and the stannous chloride reaction obtainabl without difficulty.

Imperfect removal of mercury salt from an arsenical stain does not seriousl interfere with the stannous chloride reaction, although it is preferable to obtain solution as free from mercury as possible. When a solution containing mercuri chloride only is treated with stannous chloride, a precipitate, chiefly calomel, is produced, which on subsidence leaves the supernatant liquid perfectly colourless. In the presence, however, of $\frac{1}{100}$ milligramme of arsenic in 1 c.c. volume, the liquid, when it becomes clear, is seen to have a distinctly perceptible pink-brown tint.

For ordinary qualitative detection of arsenic, the apparatus employed should include some arrangement for washing the gas through lead acetate solution, and the nature of the stain produced should be determined as described. The simple and ingenious apparatus devised by Mr. C. T. Tyrer, fulfils these conditions admirably and its positive indications, confirmed by HCl and $SnCl_2$, may in all cases be relie upon. I think, however, it is possible to go further, and not only make the Gutzei test conducted at a boiling temperature a true negative test (with some slight reserva tion, such as when sulphide of arsenic is associated with certain organic substances etc.), but also to extend its principle to the approximate quantitative determination in the following sense, of traces of arsenic.

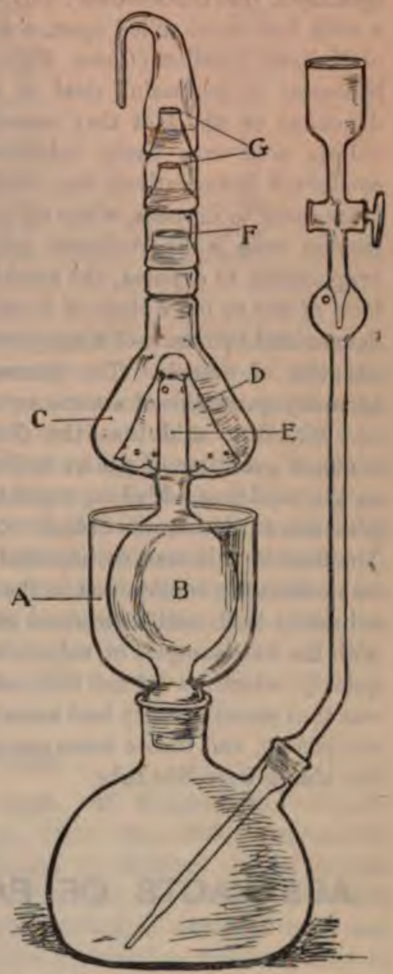
A convenient and easily recognisable standard of comparison is afforded b $\frac{1}{100}$ milligramme As_4O_6. By taking an amount of the substance under examinatio corresponding to any standard limit of arsenical impurity agreed upon for that sub stance (such limits, it is to be hoped, will be decided upon in the near future for al the more important chemicals, food products, etc.), it appears possible to say, wit the minimum of expenditure of time and trouble, whether the limit has been exceede or not. In this way, with a limit of $\frac{1}{100}$ grain per gallon (*e.g.*, beer), the miniatur gallon of 70 c.c. would be taken for the test = 1 part in 7 millions. With 10 gramme of material $\frac{1}{100}$ milligramme arsenic = 1 part in 1 million, with 7 grammes $\frac{1}{100}$ grai per pound, with 1 gramme 1 part in 100,000, etc. I think the Gutzeit test, for rapi approximately quantitative determinations, such as the foregoing, has very consider able possibilities.

The apparatus figured opposite has been devised with the view of combining in single piece the condenser, wash-bottle, 5-millimetre tubes, etc. It is constructe entirely of glass, with ground joints as shown, and admits of rapid and effectiv cleansing by rinsing each part with a stream of water from the laboratory tap.

The sketch is almost self-explanatory. The tap-funnel shown on the right-han side is for delivering the acid slowly into the flask; the glass cup above the flas is filled with water, taking the place of a condenser, and the next bulb contain 10 per cent. lead acetate solution. The flow of acid into the gently-boiling liqui should be adjusted by the stopcock to a regular succession of drops, in order t maintain a uniform evolution of gas and a slight pressure in the apparatus equ to a column of liquid 5 or 6 inches high in the vertical delivery-tube. With thi apparatus a third tube is provided, bearing a disc of mercuric paper 20 mm. i diameter, for use when the quantity of arsenic is considerable, as indicated by th immediate staining of the lowest disc.

Marsh's test has been shown by Mr. Otto Hehner to be sensitive to $\frac{1}{1000}$ of a milligramme of arsenic; with Gutzeit's the limit (capable of confirmation) is reached at $\frac{1}{100}$. This modified method has only been applied to the detection of arsenic in chemical products, syrup, glucose, beer, etc.; and to these classes of substances alone are the foregoing statements intended to apply.

I have ventured to bring these results before the members of this Society, as my own experience with the modifications described has been so favourable that I felt they were, at all events, worthy of trial by others. Without any regard to the admitted facility of application of the Gutzeit test, it appears to me to be a distinct advantage to have at one's disposal an alternative and approximately quantitative method which will, with a moderate quantity of material, rapidly indicate whether the arsenical impurity of a given substance be near or far removed from the limit agreed upon for that particular substance. I also trust that the use of the hydrochloric acid and stannous chloride identification reactions will prevent the recurrence of those apparently inexplicable discrepancies between the results of the Marsh-Berzelius and Gutzeit methods which have been a not uncommon experience in the past.



A, Cup containing water; B, watch-shaped bulb, acting as condenser for aqueous vapour; D, E, inverted funnel, for washing the gas through lead acetate solution in C; F, lead acetate paper disc; G, 5 mm. mercuric chloride paper discs, fastened gas-tight at their edges to the contracted extremities of the glass-tubes at G.

DISCUSSION.

Mr. CHARLES T. TYRER thought that in Mr. Bird's paper rather too much stress had been laid upon the quantitative side of the Gutzeit test. The value of the test for qualitative purposes, however, when used with the addition of a lead acetate bulb, had been recently demonstrated by Mr. William Thomson. He was afraid he could not appreciate the advantage of carrying out either Gutzeit's or Marsh's test at boiling temperature. It seemed to him that it would be quite impossible to obtain concordant results, for an increase or decrease in the rate of heating must surely give rise to discrepancies. The rate of evolution of the gas could certainly be controlled better in the cold than at a high temperature. When a statement as to the quantity of arsenic present had to be made on the basis of a stain, or of an unweighable deposit or mirror, it seemed to him that such statement ought only to be made with the qualification "approximately." The necessity of relying upon a stain or mirror, the intensity of which might vary according to the conditions and the duration of the operation, was unfortunate; but where, in determining minute quantities of arsenic, a stain had to be relied upon, a method recently described by Dr. Albert Atterburg, of Kalmar, Sweden (*Chem. Zeitung*, vol. xxv., No. 25, p. 264) seemed capable of becoming a successful rival of the present quantitative methods. The process depended on the fact that arsenious acid, when heated with concentrated hydrochloric acid, was easily volatilized, the arsenious chloride vapour being readily condensed quantitatively into water. This solution was treated with nitric acid and evaporated to dryness, when all the arsenic was left in the residue. On treating the residue with a hydrochloric acid solution of sodium hypophosphite, and again evaporating to dryness, the arsenic was deposited on the bottom of the basin in the form of one or more rings of a colour varying from gray to black, the quantity being determined by means of a comparison experiment made simultaneously with a known quantity of arsenic. The process was capable of determining with approximate accuracy quantities of arsenic as small as 0·01 milligramme.

Mr. BIRD said that the difficulty suggested by Mr. Tyrer of maintaining a constant evolution of gas at boiling temperature did not really occur in the process as the acid was added in regulated drops, and the gas generated under a certain pressure, so that it was obliged to force a passage through the substance of paper. The flow of acid was so adjusted that a column of liquid about 4 to 6 inches high was constantly maintained in the delivery tube. He had not meant to infer that the sulphuric acid itself contained sulphuretted hydrogen. What he meant was that with the best samples of sulphuric acid the gas evolved discoloured lead paper very quickly, which he did not find to be the case with hydrochloric acid. His contention was that sometimes dry lead acetate paper did not keep back the sulphuretted hydrogen completely, and then a trace came over and modified the appearance of the mirror in the Marsh-Berzelius tube.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

The Composition of Coffee from the Grand Comoro Island. **G. Bertrand** (*Bull. Soc. Chim.*, 1901, xxv., 379-381.)—The coffee plant discovered by Humblot i indigenous to this island. It was at first regarded as a distinct species, and named provisionally by Baillon *C. Humblotiana.* Froehner, however, does not admit this and regards it as merely a variety of *C. Arabica.*

It is distinguished by the remarkable fact that it contains no caffeine, the autho obtaining no trace of that alkaloid on extracting 1 kilogramme of the powdered berries. He is therefore inclined to agree with Baillon in regarding this coffee-plant as a separate species. As evidence that the climate and soil of Grand Comoro hav not influenced the composition of the plant, he gives the following analyses of the berries of *C. Humblotiana* and of *C. Arabica* which had been grown in the island:

	Coffea Humblotiana. Per Cent.	*Coffea Arabica.* Per Cent.
Water	11·64	9·74
Ethereal extract	10·68	5·76
Alcoholic extract	8·42	12·10
Reducing sugars	0·80	0·29
Non-reducing sugars	4·20	4·86
Total nitrogen	1·50	1·95
Ash	2·80	3·66
Caffeine	0·00	1·31

C. A. M.

Determination of Scammony for Commercial Purposes. P. L. Aslanoglou. (*Chem. News*, vol. lxxxiii., p. 146.)—A weighed quantity of scammony is extracted by warming with ether, several times in succession, each extract being filtered through cotton-wool. After washing the filter with warm ether the filtrate is treated with turpentine and left to stand for some hours, whereupon scammony, being insoluble in turpentine, comes down as an oily precipitate, leaving any accompanying gum-resins behind in solution. The precipitate is washed with turpentine, evaporated gently on the water-bath, and weighed. The insoluble earthy matter is estimated by drying the filter and contents, followed by incineration and weighing, the filter ash being deducted. The foreign gum-resins are found by evaporating the ether-turpentine solution.

C. S.

[This method seems to present some difficulties.—W. C.]

ORGANIC ANALYSIS.

Preparation of Chemically Pure Oxalic Acid. O. Schmatolla. (*Apoth. Zeit.*, 1901, xvi., 194; through *Chem. Zeit. Rep.*, 1901, 95.)—Fifty grammes of commercial oxalic acid are dissolved in 100 grammes of absolute alcohol on the water-bath, and the solution is allowed to cool and settle. It is filtered, shaken with 2 or 3 drops of 1 : 2 sulphuric acid, put aside overnight, decanted from any deposit of sulphates, the spirit distilled off, the residue dissolved in 200 or 300 c.c. of water, and the liquid left for several hours in the cold. Any impurities separating as a heavy oil are removed, the solution is filtered, allowed to crystallize, the crystals being finally dried at 35° or 40° C., and then over calcium chloride in a dessicator.

F. H. L.

On the Breinl Reaction for the Detection of Sesamé Oil. A. J. J. Vandevelde. (*Bull. de l'Ass. belge.*, 1900, xiv., 222-226.)—In Breinl's reaction a 3 per cent. alcoholic solution of the aromatic aldehydes *p*-oxybenzaldehyde, vanillin, piperonal or heliotropin is used instead of furfural. One c.c. of the reagent is added to 10 c.c. of the oil, and the mixture shaken with 10 c.c. of concentrated hydrochloric acid, a violet coloration being obtained in the presence of sesamé oil.

The author finds that olive, clove, linseed, almond, arachis, poppy, colza, c and neatsfoot oil give slight colorations in this test, whilst a very pronounced c is given by liver and rosin oils.

Comparative experiments have also shown that the Badouin reaction is pronounced and lasting than Breinl's reaction. Breinl proposed to use a petr spirit solution of sesamé oil, but the author states that petroleum spirit and h chloric acid give violet colorations with vanillin and piperonal by themselves.

The higher the boiling-point of the petroleum spirit the more pronounced reaction, and an intense reddish-violet colour is obtained with petroleum boiling at 130° C. Moreover, vanillin hydrochloride and piperonal hydroch have strong dyeing properties, and wool immersed in a solution of them is violet. The colour cannot be extracted with any of the ordinary solvents. treatment with ammonia it changes to red. C. A.

The Purification of Phloroglucinol. G. S. Fraps. (*Amer. Chem. J* xxiv., 270.)—In the determination of pentosans by distillation with hydrochlori the phloroglucinol used for precipitating the furfural must be free from diresor By the following method a product is obtained giving results agreeing well those given by Merck's phloroglucinol, which is very expensive: 11 grammes p glucinol are gradually added, with constant stirring, to 300 c.c. hot hydroc acid specific gravity 1·06. The hot solution is then poured into enough of the hydrochloric acid to make the volume up to 1,500 c.c., and the difficultly s diresorcinol allowed to crystallize out. The solution is filtered immediately use. A. G.

The Essential Oil of Kuskus Grass. E. Theulier. (*Bull. Soc. Chim.*, xxv., 454-465.)—This oil is obtained from the roots of *Andropogon muricatus*, is popularly known as *Kuskus*, or Indian dog-grass. The oil extracted in In usually obtained by distilling a mixture of the grass and Santal-wood, and but of the pure product is exported.

The chief source of the oil sent to Europe is the Isle of Réunion. The from the dried roots usually varies from 0·1 to 0·8 per cent., though in excep cases it may be as much as 3 per cent.

The author has examined two specimens of the oil, the first of whi extracted himself, whilst the second was a sample of Bourbon oil. These ga following results:

	I.	II.
Specific gravity at 20° C.	1·0091	0·986
Optical rotation at 20° C.	+35°10′	+28°
Solubility in 80 per cent. alcohol ...	1 in 1·5	1 in 1·5
Total saponification value	44·40	18·28
Saponification value of esters	11·90	12·12
Acid value	32·48	6·16

On distillation under reduced pressure (25 millimetres) the following fractions were obtained from 200 grammes of the oils:

Fractions.		Specific Gravity.	Rotation at 20° C.	Solubility in Alcohol, 80 per cent.	Total Sap. Value.	Sap. Value of Esters.	Acid Value.
C°.	Grammes.						
I. (*a*) 146—155	15·50	<1	−3°10′	Insol.	22·4	4·2	18·2
(*b*) 155—170	35·80	At 21° C. = 0·9735	+2°18′	,,	14·0	7·0	7·0
(*c*) 170—185	50·15	At 20° C. = 1·0055	+19°54′	1 in 2	22·4	14·0	8·4
(*d*) 185—205	34·10	At 21° C. = 1·0201	+36°20′	1 in 1·5	56·0	42·0	14·0
(*e*) 205—210	15·20	—	+34°8′	1 in 1	98·0	75·6	22·4
(*f*) Residue	19·25						
			At 21° C.				
II. (*a*) 144—154	30·10	At 19° C. = 0·9515	−5°50′	Insol.	16·8	2·8	14·0
(*b*) 154—167	67·50	0·9683	+0°22′	,,	5·4	1·6	3·8
(*c*) 167—177	60·60	At 21° C. = 0·9992	+31°30′	1 in 1	14·0	4·2	9·8
(*d*) 177—185	30·80	At 22° C. = 1·0001	+51°12′	,,	25·2	9·8	15·4
(*e*) Residue	11·00						

C. A. M.

Examination of Commercial Diastase for Liquefying Power. **G. Barth.** (*Zeits. angew. Chem.*, 1901, xiv., 368.)—This article contains an account of the methods adopted for, and the results obtained on, examining eight different samples of German and French diastase. The diastatic power was determined by the Lintner process (*cf.* ANALYST, 1896, xxi., 124), and the liquefying power was estimated by the following method, also due to Lintner: Ten grammes of air-dried potato starch are mixed with 100 c.c. of water, and kept as a uniform cream by repeated agitation. With the aid of a wide-ended pipette, 10 c.c. of the cream are measured out into each of a series of ten test-tubes, and to the tubes are added increasing quantities (from 0·1 to 1·0 c.c.) of the diastase solution. They are next placed in a water-bath at 70° C., shaking constantly before and during gelatinization. When the contents have become solid, the tubes are put into another water-bath maintained at the temperature at which liquefaction is to be observed, and they are finally examined visually at intervals of ten minutes. In this manner the products may be divided into six classes according to their appearance: (1) The mass will not move on inverting the tube; (2) it moves with difficulty; (3) it moves easily; (4) it yields large bubbles on shaking; (5) it yields a persistent froth; and (6) the liquid becomes clear and deposits starch-cellulose.

The effect of the various preparations upon 10 per cent. starch-paste was also examined. Thirty grammes of starch were mixed with 280 c.c. of cold water and 10 c.c. of a liquid containing 0·25 gramme of diastase. This was agitated, and raised to 70° C. for liquefaction. After cooling to 62°, another 10 c.c. of diastase solution was added, and the whole was kept stirred between 60° and 62°, until iodine showed that all starch had disappeared. The liquid was filtered, and the dry substance and the specific rotatory power at 17·5° determined. The reducing power of the samples was ascertained by carrying out Wein's maltose estimation. Dextrose in the products of reaction was tested for by heating 20 c.c. of the 10 per cent. solution with 2 grammes of phenylhydrazine and 2·2 grammes of 5 per cent. acetic acid on the water-bath for seventy-five minutes, dextrosazone then separating as fine yellow needles.

The results are given in the annexed table. It will be seen that there is no connection between the diastatic and the liquefying power of the materials.

	Taka.	Witte.	Merck.	Remy.	Defresne.	Jeune.	Billault.	Chaix.
Water	10·35	10·47	7·75	11·64	10·21	5·17		
Dry substance ...	89·65	89·53	92·25	88·36	89·79	94·83		
Ash	33·51	5·64	17·37	2·11	7·30	1·49		
Nitrogen, Kjeldahl	2·65	13·09	6·77	0·84	5·05	1·02	0·83	0·31
Proteid, N × 6·25	16·56	81·81	42·31	5·25	31·56	6·38	5·19	1·93
Diastatic value	8·63	27·4	11·5	—	Very small	Very small		
Diastase used per 100 c.c. ...	0·255	0·0832	0·1737	—	0·204	0·5088		
Specific rotatory power	146·7	156·1	151·7					
Dextrose produced	Much	None	None					
Iodine reaction of diastase solutions	None	Very faint	Very faint	Blue colour	Blue colour	Red colour	Blue colour	Blue colour
Liquefying power at 65° C. ...	Strong	Strong	Very strong					
Diastase used per 100 c.c. ...	0·20	0·20	0·1993					

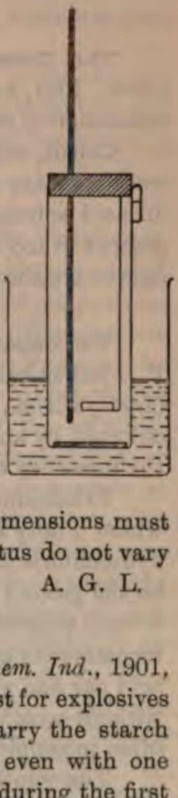
F. H. L.

Determination of Urea in Urine. A. Braunstein. (*Zeits. physiol. Chem.*, 1900, xxxi., 381; through *Chem. Zeit. Rep.*, 1901, 62.)—The author finds that the Mörner-Sjöqvist method of estimating urea is useless when applied to liquids containing hippuric acid, Bödtker's statement to the contrary notwithstanding. He gives the following process, which is simple and accurate: 5 c.c. of urine are treated with 5 c.c. of a mixture of barium chloride and hydroxide, and 100 c.c. of alcohol-ether (2 : 1) are added. After standing all night in a closed flask, the liquid is filtered, the precipitate washed six or seven times with 50 c.c. of the alcohol-ether, and the filtrate evaporated at a temperature not exceeding 55° C. When the solvents have disappeared, a little water and a pinch of magnesium oxide are introduced, and the whole is again evaporated till the vapours are no longer alkaline. The residual liquid (10 or 15 c.c.) is run into a small Erlenmeyer flask, in which 10 grammes of crystalline phosphoric acid have been placed, and heated in an air-bath for four and a half hours at a constant temperature of 140° to 145° C. When cold, the mass is taken up in water, brought into a Kjeldahl flask, made alkaline with potassium hydroxide, and the ammonia distilled into standard sulphuric acid. The liquid does not become warm when made alkaline, so there is no fear of losing ammonia.

F. H. L.

The Estimation of Hippuric Acid. W. A. Cates. (*Chem. News*, vol. lxxxiii., p. 121.)—The latter part of the Bunge and Schmiedeberg method may be shortened by taking up with hot water the residue from the petroleum ether extraction, and titrating direct with decinormal caustic soda, in presence of phenolphthalein. The end point is very satisfactory, although urinary pigment is present; and the results agree well with those of the gravimetric method. C. S.

A Comparative Method for Determining the Fusing-points of Asphalts. Charles F. Mabery and Otto J. Sieplein. (*Journ. Amer. Chem. Soc.*, xxiii., 16.)—Since the fusing-points of asphalts vary with the conditions of heating, the following standard method, which yields good comparative results, is proposed by the authors: In a glycerin bath a moderate-sized beaker is placed containing a narrow beaker closed with a cork carrying a thermometer. Through the cork is also inserted a strip of metal, ½ inch wide, extending to within ½ an inch of the bottom of the beaker, where it is bent at right angles, the narrow corners being also bent upwards. On the bend in the metal the section of asphalt is supported, being pressed down on the sharp corners. The whole is gradually heated and kept at some convenient temperature (180° C.), and the narrow beaker placed in it. The fusing-point is taken as the point at which the softened sample just touches the bottom of the beaker on either side of the bend. A metal disc placed on the bottom of the beaker greatly facilitates removal of the specimen.



The dimensions of the apparatus used were as follows: Outside beaker, width 2½ inches, height 3½ inches; inside beaker, width 1¾ inches, height 4⅛ inches; length of lower bend of support ¾ inch; width of metal support ½ inch; distance of specimen from false bottom of beaker ½ inch; distance of thermometer from specimen ⅛ inch; size of specimen 1 by ½ by ⅛ inch. The last four dimensions must evidently always be kept constant. Results obtained with this apparatus do not vary more than 2° or 3° at most, and generally there is no variation. A. G. L.

The Heat Test for Explosives. W. Cullen. (*Journ. Soc. Chem. Ind.*, 1901, xx., 8.)—The frequent irregularities in the results given by the heat test for explosives are due in great measure to the use of different grades of paper to carry the starch and potassium iodide reagents on which the test depends. Moreover, even with one particular make of paper sensitiveness diminishes with age, especially during the first six weeks after the material is prepared; but from that date onwards its indications become much more constant. Cullen therefore suggests that some arrangement should be come to among manufacturers and others to use only one kind of paper (as is done by the War Office), and to have it prepared in batches sufficient to last several years by some central authority. The Government paper is like ordinary thin blotting, 0·0082 inch thick on an average, and weighing in the air-dry state 75 grammes per square metre. As an alternative he proposes—and the desirability of this was emphasized by speakers in the subsequent discussion—to seek some substance other than paper to carry the reagents, thus avoiding the presence of adventitious matter which may affect the indications of the test. F. H. L.

Fluorescein as an Indicator. H. Zellner. (*Pharm. Zeit.*, 1901, xlvi., 100; through *Chem. Zeit. Rep.*, 1901, 40.)—The author strongly recommends fluorescein as an indicator, especially in determining ammonia when the reaction is extremely sharp. It is often indispensable in titrating dark or coloured liquids. Carbonates do not interfere. 0·4 gramme should be dissolved in 50 c.c. of alcohol and diluted with 30 c.c. of water. F. H. L.

INORGANIC ANALYSIS.

The Determination of Copper in Pyrites. O. N. Heidenreich. (*Zeits. anal. Chem.*, 1901, xxxiv., 15-17.)—The author recommends the use of aluminium for the separation of the copper from iron previous to electrolysis.

Cobalt, nickel and zinc, when present in small quantity, do not affect the results, since they are only partially precipitated, and are not separated by electrolysis from an acid solution. Lead is deposited as peroxide on the positive pole. When not present in too large amount it also is without influence on the results. Satisfactory figures are shown in the tables giving the results of test experiments. C. A. M.

The Separation of Arsenic. M. Rohmer. (*Berichte*, 1901, xxxiv., 33.)—Since E. Fischer based a method of separating arsenic on the volatility of its chloride (*Liebig's Ann.*, ccviii., 182), various attempts have been made to replace the ferrous chloride by another reducing agent, so as to overcome the difficulties introduced by the necessity of using so much of that salt.

Friedheim and Michaelis (ANALYST, xx., 235) recommended methyl alcohol, whilst Piloty and Stock (ANALYST, xxii., 297) distilled the arsenic in a current of hydrochloric acid gas and hydrogen sulphide. The author objects to the first method on the ground that several distillations are necessary, and to the second because the arsenic sulphide must be freed from sulphur before weighing. The method which he recommends consists in distilling the arsenical solution in a current of hydrochloric acid gas and sulphur dioxide in the presence of a small quantity of hydrobromic acid, without which, as Bunsen has shown (*Liebig's Ann.*, cxcii.), the reduction is slow and often incomplete.

The solution containing the arsenic is placed in a 500 c.c. flask with a long neck, and sufficient concentrated hydrochloric acid added to fill the flask to about a third of its volume. One gramme of hydrobromic acid is then introduced (about 1·5 grammes of potassium bromide or 1 gramme of bromine and sulphurous acid).

The flask is inclined at an angle of 45° and closed with a rubber cork, through which pass the delivery tube, and a tube which reaches nearly to the bottom of the flask. The latter is connected with a T-piece, through which a rapid current of hydrochloric acid gas and a slow current of sulphur dioxide are introduced. The delivery tube is conducted to the bottom of a litre flask containing 300 c.c. of water, which is cooled in a freezing mixture.

The distillation is continued for about forty-five minutes, until the residue is only about 40 c.c. Under these conditions, 0·15 gramme of arsenic is completely expelled by one distillation.

For a gravimetric determination the distillate is boiled for twenty minutes under a Liebig's condenser to expel the sulphur dioxide, a current of carbon dioxide being meanwhile introduced through a long tube passing through the inner tube of the cooler. The arsenic is then precipitated as sulphide, and weighed with the usual precautions.

It can also be determined volumetrically by boiling an aliquot portion of the distillate, previously diluted with twice its volume of water, in a beaker covered with a clock-glass, and, after removal of the sulphur dioxide, titrating the arsenious acid with $\frac{N}{10}$ iodine solution.

Antimony, when present, is left in the distillation flask, and can be determined as sulphide after removal of the sulphur dioxide and dilution. The precipitate can be completely freed from small quantities of sulphur by heating it for two hours at 225° to 230° C. in an atmosphere of carbon dioxide. C. A. M.

On the Separation of Zinc from Nickel (and Cobalt). F. P. Treadwell. (*Zeit. anorg. Chem.*, xxvi., 104.)—In Zimmermann's method the zinc is precipitated from a nearly neutral solution of the chlorides, to which an excess of ammonium thiocyanate has been added, by means of hydrogen sulphide at 70° C. Experiments were carried out to see whether the ammonium thiocyanate could be replaced by other salts of strong acids. To the solution containing an amount of oxides of zinc and nickel (as sulphates or chlorides) equivalent to $\frac{1}{4}$ per cent. of the solution, 8 or 10 drops of binormal hydrochloric acid and 2 per cent. of ammonium sulphate or chloride were added, and hydrogen sulphide was then passed in to saturation at 60° C. The precipitate was filtered, washed with hydrogen sulphide water containing 2 per cent. of the ammonium salt and a few drops of mineral acid, and the zinc determined as usual. Using ammonium chloride and sulphate, the results obtained were as good as those given by ammonium thiocyanate. The separation of zinc sulphide is also stated to be complete in the presence of potassium chloride or sulphate. A. G. L.

Detection and Estimation of Minute Quantities of Manganese. H. Marshall. (*Chem. News*, vol. lxxxiii., p. 76.)—The author finds that small quantities of manganese can be detected by gently warming the solution with potassium or ammonium persulphate (the former being generally preferable) in presence of sulphuric or nitric acid, and one drop of dilute silver nitrate solution. A pink coloration is developed, even when the final volume of liquid (about $\frac{1}{2}$ c.c.) contains only 0·001 milligramme of manganese. The reaction is apparently due to the formation and decomposition of silver peroxide derived from the persulphate. It may be utilized as a colorimetric test, the standard permanganate solution used for comparison being reduced for storage to prevent loss by deposition, and re-oxidized before use. C. S.

INSTITUTE OF CHEMISTRY OF GREAT BRITAIN AND IRELAND.

We note that the first examination in the branch of Biological Chemistry will be hel at the Institute of Chemistry in October next, Professor Adrian J. Brown being th examiner. The precise date is not yet fixed. The examination is open to an candidate whose application for admission to the final examination has been accepte by the Council, and to any candidate who has passed the intermediate examinatio of the Institute. It will extend over at least four days, and may be theoretica practical, written, and oral. The syllabus is as follows: Biological chemistry, wit special reference to the chemistry and bacteriology of foods, water, sewage, an effluents, and the practical applications of biological chemistry to industrie Candidates presenting themselves in this or any other branch of the final examinatio will be expected to possess a general knowledge of all branches of chemistry, addition to a thorough knowledge of the particular branch selected, and they must familiar with the use of such scientific instruments as are commonly found chemical laboratories. Any candidate for the final examination may present a thes upon any chemical subject to which he has paid special attention, and such thesis, approved by the examiners, may be taken as part of the examination. Candidat are also at liberty to submit note-books containing records of any work done by the which have not already been inspected by the examiners. The contents of note-boo will be taken into consideration.

The course of study recommended is: A course of forty to fifty lectures the morphology and physiology of micro-organisms. Practical work to includ (*a*) Microscopy, the preparation, staining, mounting, drawing, and recognition specimens; (*b*) the preparation and study of pure cultures; (*c*) the conduct fermentation experiments and the study of chemical changes brought about bacteria, moulds, yeast, etc. Examinations in the branch of biological chemistry the final examination will be held annually in October, while the examinations in t other branches will be held in January and July as heretofore.

REVIEW.

A Short Manual of Inorganic Chemistry. By A. Dupré, Ph.D., and H. Wils Hake, Ph.D. Charles Griffin and Co., London. Price 9s.

This well-known and excellent work has now reached a third edition. It h been thoroughly revised and brought up to date, and, while retaining the ma features of the former editions, has undergone considerable alteration, chiefly occ sioned by the arrangement of the bulk of the matter according to the periodic syste of classification, and this has necessitated the rewriting of a considerable portion the work. The general appearance of the book is to a considerable extent mar by the printing, which in places might have been much better, and does not nea come up to the usual standard of the firm from which it emanates. W. J. S

ERRATUM.

This vol., p. 138, line 10 from bottom, *for* "300 c.c." *read* "30 c.c."

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THE ANALYST.

AUGUST, 1901.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE AERATION TEST FOR SEWAGE EFFLUENTS.

BY SAMUEL RIDEAL, D.SC. LOND., F.I.C.

(*Read at the Meeting, April* 3, 1901.)

THE value of periodical determinations of the changes in dissolved oxygen being now generally admitted, a uniform method of applying the test is under consideration. Many outdoor circumstances, such as space and time, soil, life, flow, and wind, cannot be imitated in the laboratory, so that the test so applied must always be to some extent an artificial one.

The agencies at work in the natural purification are: (1) Anaerobic and semi-anaerobic bacteria effecting hydrolytic or resolving changes without the presence of dissolved oxygen; (2) aerobic organisms, requiring it and consuming it in their life; (3) algæ and water-plants, living partially on the carbonic acid excreted by the bacteria and liberating oxygen required by the aerobic forms. This third class demands the presence of light, the other two work best in the dark, so that the cycle of changes depends on the action of the three classes, but cannot be well carried on in the same area. In the deeper layers of ponds and rivers there is very little free oxygen, and the changes are mainly anaerobic. In the upper layers oxygen is rapidly absorbed from the atmosphere. If the organic matter, however, be so large in quantity that the absorbed oxygen is not sufficient for the aerobic bacteria, it can be supplemented by the decomposition of nitrates, or by the oxygen disengaged by green algæ. A healthy condition would be that the balance of dissolved oxygen should be maintained, so that the three classes of organisms could work in their respective zones.

In examining effluents as to whether their quality is sufficiently good to allow them to be mixed with the local river water without putrescence, both the closed and the open tests are in use.

The former, introduced by Dr. Dupré in 1885, and now regularly worked by Mr. Scudder at Manchester, preserves the liquid in a full bottle, and determines the dissolved oxygen at the beginning and at intervals of one, two, three or more days.

This fermentation resembles that at the bottom of rivers and of deep ponds. But dissolved oxygen in saturated water only amounts to about 1 part by weight 100,000, so could not be sufficient to oxidize the organic matter of mixed liq which, exposed to the air, would rapidly improve. I have just determined the behav of a town sewage as to dissolved oxygen under open and closed conditions, before after passing through a septic tank.

	Dissolved Oxygen in C.c. per Litre.	
	Raw Sewage.	Tank Efflue
Kept six days in closed full bottles	0·59	0·43
Exposed to air in a layer 1 foot deep :		
Twenty-four hours	1·81	2·06
Forty-eight hours	3·02	4·74

The odour in the closed bottle increased ; in the second case of exposure to it was very slight after twenty-four hours, and none after forty-eight hours. Un natural conditions, therefore, the sewage would have become inoffensive.

More artificial is the system of conducting the closed process in the dark. All Levy suggested that in this way the action of bacteria could be discriminated f that of green algæ, which require light for evolving oxygen. Having ascertained original free oxygen, he enclosed the water in a full bottle placed in the dark at 33 for forty-eight hours and determined the oxygen left; the loss, divided by the orig amount, was called the "coefficient of alterability," which he considered a measur the quantity of bacteria. It, however, only regards a section even of the bacte and does not relate to natural conditions. The test is systematically carried out Paris.

The Open Process.—The depth and breadth of bodies of water, the circulatio the fluid, the temperature, and the amount of light, have all an influence on natural purification, and vary greatly at different places and times. The characte the organisms will also vary.

Trials made in the laboratory with small quantities give valuable informati but the conditions should be stated, and for purposes of comparison a stand procedure is needed. Dibdin and Thudichum (*J.S.C.I.*, June 30, 1900) allo mixture of one part of effluent and one of fully aerated water to stand in an o vessel *of diameter equal to the depth of the liquid*, and determine the dissolved oxy daily for a certain number of days; it should not fall below 50 per cent. of maximum possible at the temperature. "Whenever the aeration of the water of Thames was found to exceed this minimum there was no suggestion of nuisance, fish appeared in the areas within which they could not live during other periods."

In regard to the "fish test," Duncan and F. Hoppe-Seyler (*Zeit. Physiol. Che* xvii., 165) found that perch, trout, etc., could thrive in water holding 3 to 4 c.c oxygen in solution per litre, but soon died when only 1·7 c.c. was present. W reference to the absorption of oxygen by water, the same observers (*Zeit. Phys Chem.*, xvii., 147) filled tubes of different lengths with boiled water and exposed th to the atmosphere for varying periods, noting the temperature and barome pressure. From the results they calculated tables of the diffusion of the gases i the water at different depths below the surface. The total amount of gas absor increased with the duration of exposure, but the daily increment in the amount absorbed gradually diminished.

Mr. Dibdin also remarked (*loc. cit.*): "If the aeration fell below 50 per cent., it showed that a quantity of organic matter was present on which a number of aerobic organisms were being maintained, with the result that they were absorbing oxygen quicker than it could be reabsorbed by the water from the atmosphere. But if 50 per cent. or over of aeration were steadily maintained, and if the percentage steadily arose, it was clear that the sample was progressing towards absolute purification." From the results of London County Council experiments, he concluded that oxygen-free water when suddenly exposed to the air took up during the first hour about 15 per cent. of the maximum absorption, and that as the quantity of oxygen absorbed in the water increased the rate of absorption decreased, "so that at some point a balance would be reached." But sewage, although nearly free from oxygen, is not comparable with boiled distilled water. When the oxygen as it dissolves is taken up by substances in the water, the rate of absorption will remain rapid. To imitate approximately the natural circumstances, I have mixed boiled water with ammonio-cuprous chloride and exposed it to air under various conditions, judging the absorption of oxygen by the tint of blue produced.

I. Three tubes of equal diameter (½ inch), and respectively 18, 12 and 6 inches long. After twenty hours the colours were inversely as the lengths. In sixty hours they were nearly equal, and all about half saturated. After eighty-four hours they were equal, but still short of saturation.

II. The amount of cuprous salt was made equivalent to the maximum dissolved oxygen at the temperature. The tubes were 12 inches long and 1 inch wide. The first tube gave:

Hours	1	2	3	4	5	22
Oxygen, c.c. per litre	1·0	1·5	2·5	3·0	3·0	saturated

A second tube, probably owing to more constant conditions, took a longer time to reach the maximum. It gave in one hour, 1·7 c.c. oxygen; two hours, 2·6; three hours, 3·0. After this the increase of colour for some hours was so slow as not to be measurable. In eighteen hours it was equal to 3·5 c.c. oxygen; the upper 7 inches had nearly reached saturation blue, the lower 5 were almost colourless. Twenty-six hours, 4 c.c. oxygen; thirty-eight hours, colour 9 inches down, oxygen 5 c.c. Saturation reached in fifty hours.

III. Same as last, but with wider vessels, depth and width 3 inches.

Hours	1	2	3	4	20
Oxygen, c.c. per litre	1·5	2·5	2·7	3·0	saturated

In moderately shallow layers the results seem to depend less upon depth and width and amount of oxidizable matter than upon convection currects and vibration of the liquid. In the next series of experiments actual sewages were mixed with tap-water in equal proportions, placed in cylinders of varying sizes, and exposed to air and diffused light at ordinary temperatures. The liquids used were:

A. In experiments 1 and 2, a town effluent from sewage works, containing 4 parts of chlorine per 100,000.

B. Experiments 3, 4 and 5, a town sewage with 10 parts Cl.

C. Experiments 6 to 10, a foul-smelling sewage with dark earthy sediment; chlorine, 5·5 parts.

The physical conditions were:

No.	Depth, Inches.	Width, Inches.	Surface Area, Square Inches.	Volume.		Relative Volumes, Approximate.	Surface to Volume, Approximate.
				Cubic Inches.	C.c.		
1 and 6	6	2	3·14	18·85	309	1	1 : 6
2 and 7	12	2	3·14	37·7	618	2	1 : 12
3 and 8	6	5	19·6	117·6	1,929	6	1 : 6
4 and 9	6	2¾	5·9	35·4	580	2	1 : 6
5 and 10	2	10	78·6	157·2	2,578	8	1 : 2

Dissolved oxygens:

No.	C.c. per Litre.			Percentage of Saturation.		
	24 Hours.	60 Hours.	120 Hours.	24 Hours.	60 Hours.	120 Hours.
A. 1.	5·49	5·35	—	89	81	
2.	4·8	5·2	—	77·5	79	
B. 3.	3·3	6·1	—	53	92·5	
4.	3·35	6·05	—	54	92	
5.	4·2	6·25	—	68	95	
Tap-water	6·2	6·6	—	100	100	
		72 hours			72 hours	
C. 6.	2·25	3·13	—	35·4	52	
7.	1·46	2·36	—	23	39	
8.	3·0	4·20	5·64	47	70	97
9.	2·68	4·08	—	42	67	
10.	3·95	5·25	5·58	62	87	96
Tap-water	6·36	6·02	5·8	100	100	100

After sixty hours Nos. 1 and 2 were nearly colourless, and had scarcely any odour; trace of nitrite; nitrate = 1·4 parts N per 100,000.

Nos. 3, 4, and 5 after sixty hours showed a yellowish tint, with floating mycelium or zooglœa, a distinct urinous odour, a heavy trace of nitrite, and no nitrate.

In series C, Nos. 6 to 10, after twenty-four hours, the settled liquid was opalescent, had a slight brownish tint and very little odour; nitrite, 0·15; nitrate, 0·368 part per 100,000.

After seventy-two hours there was no odour; the liquid was clear and nearly colourless. Nitrite, 0·05; nitrate, 0·42.

After 120 hours No. 8 contained 5·64; No. 10, 5·58; tap-water, 5·8; so that both were practically saturated. They were clear, with no odour and only a faint colour.

CONCLUSIONS.

1. The width of the sample has by itself very little influence on the aeration, provided the proportion of width to depth be great enough to allow sufficient circulation. A width of 2 inches, with a depth of 12 inches, is too narrow to satisfy this condition.

2. The depth, as would be expected, has a great effect; the amount of dissolved oxygen in large volumes of water diminishes with the distance from the surface, unless circulation interferes. Thus, in Adeney's tables the water of the reservoir contained at 5 feet depth 5·71 c.c. of O per litre, at 20 feet only 2·00, due to the action of anaerobes.

3. Too great a depth and too shallow a layer should be avoided, and the relation between surface and volume should be uniform in different experiments. In Mr. Dibdin's proposal of equal widths and depths (*J.S.C.I.*, June, 1900, p. 498), the ratio of surface to volume varies in each case.

4. The best directions for carrying out this form of the test uniformly seem to be: The samples should be kept at a uniform temperature in cylinders 6 inches deep and 3 inches wide, exposed to the air and light, but protected from dust. The volume will be about 600 c.c., and the ratio of surface to volume 1 to 6.

5. Even with uniform physical conditions, the action varies greatly with the nature of the organisms in the sewage and *in the water used for dilution.* Water from a water-supply that has been filtered is far less active than river water.

INFLUENCE OF ORGANISMS ON THE ABSORPTION.

Three samples of liquid—

- A. Water vigorously boiled in a flask for two hours;
- B. Sewage similarly boiled, with additions of boiled water to maintain the volume;
- C. Raw sewage previously kept in a closed vessel—

were exposed to air in a cool, quiet room in beakers, depth and width of liquid 3 inches, and the dissolved oxygen determined at intervals by the modified Winkler's process. As B and C contained nitrites and organic matter, previous permanganation was used in the manner I have described in a recent paper.*

No.	Hours.	Temp. C.	A. Boiled Water.			B. Boiled Sewage.			C. Raw Sewage.		
			C.c. O.	Roscoe's Maximum.	% of Saturation.	C.c. O.	Roscoe's Maximum.	% of Saturation.	C.c. O.	Roscoe's Maximum.	% of Saturation.
1	1	14·0	3·17	7·12	44·5	3·12	7·12	43·8	3·16	7·12	44·4
2	2	14·5	3·81	7·04	54·1	3·23	7·04	45·9	3·82	7·04	54·2
3	3½	15·5	4·41	6·89	64·0	4·12	6·89	59·8	4·26	6·89	61·8
4	5	14·0	4·84	7·12	68·0	4·70	7·12	66·0	4·42	7·12	62·0
5	11	14·5	5·69	7·04	80·8	5·13	7·04	72·8	4·05	7·04	57·5
6	25	14·5	6·71	7·04	95·3	6·22	7·04	88·3	3·0	7·04	42·6

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* ANALYST xxvi., p. 141.

The boiled sewage has behaved nearly in the same way as ordinary water. In the raw sewage the absorption is at first normal, but after five hours the consumption of oxygen by fermentations overpowers the absorption from the air, and the amount in solution sinks, till in twenty-five hours it is considerably below 50 per cent., so that by this datum the liquid would be condemned as putrescible.

ON ALKALINE WATERS FROM THE CHALK.

By W. W. Fisher, M.A.

(Read at the Meeting, May 1, 1901.)

The standards of purity for natural waters are necessarily based upon the ascertained composition of unpolluted samples, which will exhibit various differences according to their origin and the geological formations from which they are obtained. The extent to which waters are rendered as "hard" or "soft" depends roughly upon the quantities of calcium or magnesium compounds taken up by them, and we expect water-supplies from limestones or chalk to be hard, while those from sandstones (with some exceptions), shales, and igneous rocks, are as a rule soft in character.

Attention has, however, been repeatedly drawn to the fact that waters from deep wells in chalk in London and other places where the chalk is covered by the London clay and tertiary beds are entirely different in character from the waters obtained either from natural springs or deep wells in chalk districts where there is no such covering of impervious deposits.

The composition of a few typical chalk waters of the ordinary kind is shown in Table I., from which it appears that such waters contain from 20 to 30 grains per gallon of dissolved solid constituents. These are chiefly carbonate of lime, with a little magnesia, with small quantities of alkaline chlorides and sulphates. These waters are always hard to an extent proportional to the lime and magnesia in solution.

On the other hand, the waters obtained from the chalk beneath the London clay contain only mere traces of lime and magnesia as carbonates, while considerable quantities of alkaline salts, mainly consisting of sodium chloride, sulphate, and carbonate, are present, and these waters, consequently, are soft and alkaline.

In Table II. are given some examples of waters of this type derived from various localities, ranging from near Reading in Berkshire to Chelmsford in Essex, and in some of these waters the excess of saline constituents is very marked. The Essex sample contains 35·7 grains per gallon of common salt, the total solids being 74·8, with 7·3 grains of anhydrous sodium sulphate and a substantial amount of sodium carbonate; while the Wokingham water with 66 grains of total solids, contains 41·2 grains of common salt, with sodium sulphate and carbonate in distinct but smaller proportion. The water from Clewer, near Windsor, is similar in character. Many of the waters, it will be noticed, contain traces of free ammonia, but the proportion of nitrates is always small.

No.	Locality.	Depth in Feet.	Total Solids.	Chlorine in Chlorides.	Nitrogen in Nitrates.	Ammonia.	Albu-minoid Ammonia.	Oxygen absorbed in 3 Hours.	Hard-ness.	Remarks.
3188	Slough, Bucks	211	22·68	1·0	0·210	0·025	0·005	0		
3055	Lockinge, Berks	60	33·0	1·7	0·154	0·002	0·007	0·035		
2217	Fawley, Bucks	330	38·0	2·1	0·84	0·004	0·003	0	26·2	
2747	Henley-on-Thames, Oxon ...	280	26·04	1·1	0·329	0·003	0·003	0·003	18·0	
2473	" " " ...	100	25·2	1·0	0·301	0·003	0·004	0·005		
2777	Devizes, Wilts, well on Downs	(?)	19·04	0·8	0·035	0·003	0·003	0·010	16·0	
2808	Beaconsfield, Bucks ...	270	22·4	1·0	0·210	0·001	0·001	0		
2817	Great Missenden, Bucks ...	(?)	22·1	0·77	0·210	0·003	0·001	0·007		
3025	Amersham, Bucks, Water-works well	150	19·88	0·9	0·091	0·003	0·001	0·002		
1525	Near Goodwood, Sussex ...	55	20·4	1·5	0·147	0·003	0·001	0·007	16·7	
1350	Savernake, Wilts	(?)	26·0	1·3	0·175	0·012	0·001	0·015		
2532	Hungerford, Berks	(?)	24·9	1·0	0·315	0·003	0·006	0·007	17·5	
2397	Wantage, Berks, from Downs	—	22·4	0·8	0·154	0·003	0·005	0·008		
2198	Farnham Royal, Bucks ...	60	19·0	1·8	0·231	0·001	0·003	0·023		
2166	Near Princes Risboro', Bucks	(?)	18·5	0·77	0·357	0	0·003	0		
2543	Great Marlow, Bucks ...	260	23·2	1·0	0·259	0·001	0·001	0		

TABLE II.—WATERS FROM CHALK BELOW LONDON CLAY. (*Analysis in Grains per Gallon.*)

No.	Locality.	Depth in Feet.	Total Solids.	Chlorine in Chlorides.	Nitrogen in Nitrates.	Ammonia.	Albu-minoid Ammonia.	Oxygen absorbed in 3 Hours.	Hard-ness.	Remarks.
1539	Faversham, Kent ...	74	40·0	4·9	0·05	0·006	0·004	0·015	29·2	
2057	Aldermaston, Berks ..	260	23·0	1·1	0·04	0·008	0·003	0	13·7	Slight alkalinity. 9·1 after boiling.
1888	Peckham	259	35·0	2·6	0·014	0·021	0·004	0·005	29·6	
1878	Chelmsford, Essex ...	200	74·8	21·7	0·07	0·003	0·002	0·042	2·5	Na_2SO_4 7·3. Alkalinity 29·1, as Na_2CO_3.
1644	Chelsea	278	50·0	11·2	0·05	0·035	0·005	0·010	6·2	Alkaline.
2407	Wokingham, Berks...	408	66·0	25·1	0·05	0·003	0·003	0·003	—	Na_2SO_4. Alkalinity 18·9, as Na_2CO_3.
2261	Datchet, Berks ...	170	25·0	1·9	0·04	0·004	0·003	0·010	8·2	Alkaline.
2083	Clewer, Berks ...	300	62·0	17·9	0·05	0·039	0·003	0·019	11·5	Alkalinity 18·5, as Na_2CO_3.
3194	Hook, Hants ...	600	38·3	7·0	0·035	0·067	0·003	0	17·5	Alkaline.
2458	Wallingford, Oxon ...	(?)	37·0	2·2	0·014	0·028	0·004	0·010	—	Alkaline.

Numerous wells have been sunk into the chalk below London, and careful analyses have been made in some cases of the waters obtained, and on this account the Government wells near Trafalgar Square, another at the Royal Mint, and the deep well at Guy's Hospital, are of special interest as affording an opportunity of learning whether any changes in salinity have taken place over a period of more than fifty years. After application to H.M. Office of Works, by the kindness of Mr. Westcott, Engineer-in-charge, I was able to get a sample of the water, which on analysis yielded the results given in Table III., in which also the results of former analyses are embodied.

From these results the approximate quantities of the dissolved constituents are calculated as follows:

Trafalgar Square Well-water.

Composition in Parts per 100,000.

Total solids by evaporation dried at 100° C.	85·7
Calcium carbonate	4·29
Magnesium carbonate	3·15
Sodium carbonate (anhydrous)	18·82
Sodium sulphate (anhydrous)	28·64
Sodium chloride	27·34
Potassium chloride	2·16
Ferric oxide and phosphate	0·40
Silica	1·0
Total	85·80

A comparison of the water in 1900 with the earlier samples of 1846 and 1848 shows, as regards the total saline constituents, a diminution, but since 1857 there is no substantial change in the amount—during forty-four years. It will be seen, also, that the chlorine, sulphuric acid, lime and magnesia have undergone no important alteration for upwards of fifty years; there is a striking change in the potash between 1848 and 1857, but since that time the alkalies are practically constant in quantity.

These facts are very significant, and of great interest as indicating the permanent character of the conditions affecting the supply. Although, as will be shown, the level of the water is slowly falling, from the water being pumped out from the chalk at a rate in excess of that coming in by percolation, it might have been expected that the salinity would become less; but, judging from our present knowledge, the water will retain its characteristics for an indefinite period.

Several attempts have been made to explain the abnormal characters of the waters under consideration, the most popular, perhaps, being the suggestion of infiltration of sea-water (see Braithwaite, Proceedings Institute Civil Engineers, xiv., p. 507). The hypothesis, however, is clearly untenable, and does not account for the known facts. If such infiltration were possible in Essex, it hardly could be so in London, and still less at Wokingham. Further, the chalk is full of water under greater hydrostatic pressure from the hills north and south than from the bare chalk at Erith in the tidal river, and, so far from sea-water entering there, it is well known that strong springs of fresh water discharge from the chalk into the river in that

1846 Brande	94·4	22·3	15·9	10·6	1·0	—	2·52	1·66	(54·0	)
1848 Abel and Rowney ... (Q. J. Chem. Soc. I. 97)	99·15	17·39	16·02	14·6	1·3	{0·00 0·23	2·61	1·56	35·9	10·55
1857 Campbell ... ... (Q. J. Chem. Soc. IX. 22)	84·97	16·51	17·19	8·3	0·57	0·97	2·18	1·40	39·7	1·02
1869 Frankland ... ... (Riv. Poll. Com. VI. Report)	83·40	16·55	—	—	—	—	[5·9 =	Hardness]		
1900 W. W. Fisher ...	85·7	17·6	16·14	11·35	1·0	0·40	2·40	1·50	38·0	1·35

TABLE IV.—ALKALINE WATERS FROM LIMESTONES UNDER CLAYS. (*Analysis in Grains per Gallon.*)

No.	Locality.	Depth in feet.	Total Solids.	Chlorine in Chlorides.	Nitrogen in Nitrates.	Ammonia.	Albuminoid Ammonia.	Oxygen absorbed in 3 Hours.	Hardness.	Remarks.
2749	Newington, Oxon, below Gault Clay..	85	406	148·5	0·014	0·072	0·003	0·018	—	Alkaline = Na_2CO_3 17·5; SO_3 = 88 2; CaO trace.
3008	Warborough, Oxon, below Gault Clay...	177	545	190·7	0·049	0·252	0·006	0·044	—	Alkalinity = 12·5 Na_2CO_3; SO_3 = 106 9; CaO trace.
2991	Bourton, Berks, below Kimmeridge Clay (1899) ...	265	152	40·5	0 049	0·050	0·006	0·031	2·8	Alkalinity = 39·2 Na_2CO_3; SO_3 abundant; CaO trace.
2339	Burcote, Oxon, below Kimmeridge Clay...	190	52	5·8	0·028	0·040	0·001	0·020	1·4	Alkalinity = 21·9 Na_2CO_3.
3027	Near Bicester, Oxon, below Oxford Clay	spring	47·6	4·2	0·070	0 007	0·009	0·470	—	Alkalinity = 31·5 Na_2CO_3; contains peaty organic matter and sodium sulphate; CaO trace.
2528	Oxford, below Oxford Clay ...	250	672	173·0	0	0·101	0·045	0·394	—	Alkalinity = 28·0 Na_2CO_3; SO_3 = 201; CaO = 20; Na_2SO_4 = 350.
	Northampton, below Upper Lias Clay ...	200	61	9·9	—	0·050	—	—	—	Mr. A. C. Chapman's analysis: Na_2CO_3 = 21·70; NaCl = 16·14; Na_2SO_4 = 18·10; (CaMg)CO_3 = 3·85.

locality. On chemical grounds, too, it is clear that no mixture of sea-water wit normal chalk-water could resemble our saline chalk supplies, since the relati amounts of chlorides and sulphates are entirely different, and such a mixture coul not contain free alkaline carbonate. Further, there is no relation between th amount of salinity of any waters of this class and the distance of the well from sal water or the sea.

Dugald Campbell also argued against the sea-water theory (*Amer. Journ. Che Soc.*, ix., p. 22), and came to the conclusion that the origin of the water is not th ordinary chalk stratum, but it comes from a separate and independent source, an suggests the tertiary beds above as the source of the saline matters.

In an account of Guy's Hospital well, Professor Odling (1860), who discussed th question, favoured the explanation that the most probable source of the carbonate alkali is to be found in the decomposition of alkaline silicates, chiefly silicate of sod existing in the strata above the chalk. Indeed, he showed that readily decompose silicate of soda was an actual constituent of the sand stratum overlying the cha obtained from the borings at Guy's Hospital well. But he makes also the importa point that in a well at Vauxhall the chalk-water contains more carbonate of so than the water from the sand above, and less sulphate of soda than the latter. The is much to be said in favour of this view, but it is not entirely conclusive, since th exposed tertiary beds do not yield waters of an alkaline or saline character, but on those, like the chalk, buried beneath the London clay, and subject to similar condition

In looking for a satisfactory explanation of this question, I have been strong impressed by the fact that the alkaline waters are not peculiar to wells sunk throug London clay, but are commonly (almost universally, I may say) met with in wel which, in the counties of Oxford, Berks, or Bucks, are sunk through clay form tions into the porous limestone or other beds below. Many such instances are m with in wells sunk through gault, or Kimmeridge, or Oxford clays, and I hard know an instance to the contrary. One or two examples have come to my kno ledge of wells sunk into the porous middle lias beds between the upper and low lias clays which yield saline and alkaline waters.

In Table IV. I have given a few examples of such waters.

We are therefore led to the conclusion that the origin of the saline matters mu be sought for in the beds themselves, and that the overlying impervious clay is necessary contributing factor in the case.

Now, in the Thames Basin London clay is a very extensive thick deposit, whic from Hungerford in Berks to the east coast of Essex, extends over a distance 120 miles. The width in London is about thirty miles from north to south, and Essex rather more. The north of Kent is partly covered by a long stretch of th deposit. Obviously, no water can pass downwards through this impervious lay and the water which reaches the chalk below must enter beneath the edges of t clay, and the supply to the Trafalgar Square well comes either from the Hertfor shire chalk downs, a distance of fifteen miles underground, or those near Croyd a distance of about nine or ten miles. Below the clay the chalk and tertia beds are fully charged with water, and at the edge of the clay strong springs, f by the surplus waters, break out; such waters being of the normal type of cha supplies. Owing to the depression of the chalk in the centre, the waters have natural escape open to them, and would remain indefinitely in contact with it, and even when, owing to wells being sunk, a portion is removed by pumping, it is clear that a long time is required for the underground water to travel ten or fifteen miles. The chalk itself being remarkably compact, partly owing to the pressure from above, and more perhaps to the absence of such fissures and channels as would be produced by the ordinary action of flowing water, great resistance is offered to the passage of water through it. All the conditions favour the taking up of any soluble salts by water in contact with such chalk. It may also be pointed out that uncovered chalk, being subject to percolation for ages past, has lost the greater part of the easily-dissolved constituents, while the buried chalk, having no drainage outlets, has not been washed in the same way, and the soluble matters to a large extent remain.

These considerations appear sufficient to explain the increased proportion of chlorides and sulphates in such waters, and I hope to bring before the Society evidence of the progressive increase in saline constituents after an increasing length of underground passage of water through cornbrash under Oxford clay. The explanation of the presence of sodium carbonate, although a little more complicated, is essentially the same in principle. I have observed that as a fact nearly all chalk and limestone waters contain minute quantities of alkaline carbonates, which can be detected by the action on turmeric paper, and further traces of sodium carbonate are frequently present in chalk or limestone. In the underground journey the proportion of alkaline carbonate will gradually increase, and as it increases the calcium salts will diminish, until mere traces only are left. Neither chloride nor sulphate of calcium or magnesium will remain, and only limited quantities of carbonates. The amount of calcium carbonate in solution will ultimately depend on the amounts of carbonic acid in the water available as a solvent, so that some of the alkaline chalk waters are harder than others for this reason. In support of this view, I have examined specimens of chalk from a boring made some years ago in the Tottenham Court Road, one from a depth of 500 feet, the other from 800 feet. The cores are in the Geological Department of the University Museum, and I am indebted to the kindness of Professor Sollas for some fragments.

Both specimens contained distinct traces of alkaline carbonate, sulphate and chloride, and when extracted with redistilled water in a platinum vessel gave distinct reactions with turmeric paper. After evaporation the residue was redissolved and filtered, and the amount of sodium carbonate estimated by titration with decinormal acid was 0·03 per cent. of the weight of the chalk. The chalk from 800 feet deep was sufficiently alkaline to affect moist turmeric paper when a little of the powder was placed upon it.

An analysis of the sample of chalk from a depth of 500 feet gave the following results:

Calcium as carbonate	93·90
Silica (including soluble silica)	4·20
Iron oxide (with P_2O_5)	0·50
Magnesium carbonate	0·70
Sodium sulphate	0·28
Sodium chloride	0·20
Sodium carbonate	0·03
Water and loss	0·19
	100·00

Note.—It has been shown by Way (*Journ. Royal Agric. Soc.*, vol. xii., p. 544) that the lower chalk contains considerable amounts of silica and traces of alkaline salts.

Another specimen from a depth of 800 feet yielded alkaline carbonate in the proportion of 0·05 per cent.

The level of the water in the chalk wells below London is gradually sinking, and the Royal Commission on Metropolitan Water-supply, 1893, collected information as to the condition of many such wells (Report, Appendix, C. 4, p. 157). The depth of the Trafalgar Square well was then given as 384 feet, of which 241 feet was above the chalk, and 143 feet in the chalk. The water-level in October, 1900, was 174 feet from the surface, which is still 67 feet above the top of the chalk. The levels at various dates, according to the above report, which was prepared by Mr. A. R. Binnie, the Engineer to the London County Council, are as under: 1847, -58·1 below Ordnance datum; in 1878, -83·35; in 1888, -100·68; in 1891, -108·7; in 1900, -117·1. Mr. Westcott, the Engineer-in-charge, informs me that the water does not appear to fall so fast recently, but it would appear that the level sinks about 1 foot per annum. The water clearly cannot filter or travel through the chalk for so great a distance as fast as it is being removed by pumping, and in the future possibly the level will be still further reduced.

DISCUSSION.

Mr. DIBDIN said that the question of variations in the quality of water obtained from certain deep wells in London had been one which had exercised the minds of all water analysts, who had hitherto generally been content to accept the explanation of Mr. Dugald Campbell—that the alkalinity of these waters was due to the solution of salts in the Thanet sands. But, if that explanation were correct, he (Mr. Dibdin) could not understand why the solution should not be much greater than was generally the case. He thought that the explanation suggested by Mr. Fisher, namely, the gradual solution of the soluble salts in the chalk during the progress of the water in its filtration through the chalk from the outside areas, was altogether far more reasonable. He would have been glad if Mr. Fisher had been able to give a few further analyses of waters drawing their supply from the Thanet sand. Any moderate variations that might be found in such waters would not in the slightest degree vitiate Mr. Fisher's argument, but would show how widespread was the variation in the quantity and character of their mineral constituents. There was another well close to that at Trafalgar Square, namely, in St. James's Park, of almost identical character; and on the south side of the Thames there were several wells yielding supplies of a similar character; while further down the Thames waters occurred which appeared to be mixtures of these soft alkaline waters with the hard water.

Mr. RICHMOND said that he had recently examined a sample of water from Bourton, near Swindon, which he believed to be from practically the same source as the one from that neighbourhood referred to by Mr. Fisher. There was a supply of a similar character near Shrivenham Station, but in that case the well was only a few feet deep; and on the other side of the Great Western Railway, about a mile and a half away, where the ground rose slightly, he had found the same water from a well between 50 and 60 feet deep. On the other side of Swindon, three or four miles away, where the ground rose again, practically the same water occurred, the well in this case being about 100 feet deep. In all cases there was a large quantity of sodium sulphate, and the water was always alkaline. He had found, however, in going from Bourton to Swindon that there was a slight change in the water, the chlorine becoming less and less as one went further west. The chlorine in the Bourton water was about the same as that in Mr. Fisher's sample; but on the other side of Swindon, although the sulphuric acid kept fairly constant, the chlorine fell to about 22 grains per gallon.

Mr. W. T. **Burgess** said that in some cases the alkalinity as determined by direct titration would include a certain quantity of carbonate of lime. In order to estimate the alkalinity of water, it had been his practice for a long time, in the case of waters containing carbonate of soda, to evaporate to dryness, extract with boiling water, and determine the alkalinity of the filtrate.

Mr. **Blount** said that, before one could accept so revolutionary a hypothesis as that which had been put forward as to the cause of the alkalinity of these waters, one must be quite sure of the data upon which it was founded, and he would suggest that Mr. Fisher would add greatly to the value of the paper if he would state with precision the exact methods of analysis he preferred to employ. In particular, he would ask that the method used for determining the alkalinity should be specifically stated, and also the data on which the author had been led to assume that a given water contained so much carbonate of any sort. In his own practice in water analysis, whenever a doubtful case occurred, he found that any titration method was to be put aside, the only satisfactory method in such cases being to evaporate nearly to dryness and determine the carbonic acid gravimetrically, as one would do in the case of chalk. The assumption that the alkalinity, however carefully determined, really represented the carbonic acid present in the water, and was a proper basis on which to calculate the carbonates of the mineral constituents, was apt, he thought, to lead to erroneous conclusions. The determination of each mineral constituent by some method that was quite irrefutable was the only means, not merely of arriving at a correct analysis of a water, but also of founding satisfactorily such a very interesting and comprehensive hypothesis as that referred to.

Mr. **Hehner** said that it was often difficult to distinguish between the alkalinity produced by sodium carbonate or calcium carbonate and the alkalinity produced by magnesium salts. The waters now under consideration, however, contained rather small quantities of magnesium and calcium, so that the alkalinity would not be much affected by these, but would be due practically to sodium carbonate. The question of the presence of sodium carbonate in chalk was a very interesting one. Chalk was a marine deposit, and the sea certainly contained no sodium carbonate, because it contained large quantities of magnesium chloride, and the two were incompatible. Consequently, the chalk originally was not an alkaline deposit in the sense of containing sodium carbonate, and the question as to how it became alkaline was a difficult one to answer. Anybody who had looked at a chalk cliff on the South Coast must have noticed the manner in which the alternating deposits of chalk and flint occurred therein. He had discussed the subject with many geologists, but had never been able to obtain a feasible explanation of the fact that there occurred in the chalk at every 15 or 18 inches a well-defined layer of flint, then a layer of chalk without any flint, and so on. He did not think it would satisfy anyone to be told that during a certain era there was a deposit of chalk, that then the circumstances became such that foraminifera became less abundant and sponges predominated, these again dying out and giving place to a deposit of chalk. He had always thought that, in addition to the action of living organisms, chemical action must have played a considerable part in producing the alternation of deposits, and it was quite obvious from Mr. Fisher's observations that a chemical alteration had taken place in the chalk. Chalk contained appreciable quantities of silicates, and silicates were readily dissolved, but silica was, on the other hand, readily deposited from water. Rain-water percolating through the soil becomes saturated with silica, which readily deposits again round nuclei (spicules of sponge) and flint forms. Supposing that the chalk also contained sodium salts, then it was probable that, with water containing carbonic acid, decomposition of the silicates would take place with the formation of sodium silicate, and that through the action of this on the calcium carbonate sodium carbonate might be formed. He had always attributed the alkalinity of the water from the chalk below the London clay to the Thanet sand, or possibly to the greensand. But the explanation suggested by Mr. Fisher would be far more satisfactory if it were confirmed by an examination of the waters from other chalk districts where similar conditions existed. No doubt in this or other countries there were many beds similarly situated.

Mr. Fairley said that in certain parts of Yorkshire alkaline waters occurred very frequently, especially in the neighbourhood of coal formations, and under certain portions of the millstone grit and other formations. He was not sure whether such waters always occurred in connection with beds of clay, but the quantity of carbonate of soda which they contained was sometimes very considerable. The question had sometimes occurred to him whether this sodium carbonate might have been formed from sodium salts in some way similar or analogous to that in which sodium nitrate was formed in the nitrate beds of Chili or sodium carbonate in the natron beds of Egypt, where organic matter played some part in the changing of what was no doubt originally sodium chloride into sodium nitrate in some cases, and into sodium carbonate in others. He thought that in the case of waters like those referred to it would be interesting to look for other alkaline bases. In the colliery waters of the North of England lithia was nearly always present, though it had been reported in only a few instances. Its presence was probably attributable to the decomposition of lithia mica occurring in the coal measures. In examining water for lithia, it was impossible to obtain satisfactory results without subjecting the water to a very careful process of concentration as a preliminary to spectroscopic examination. The leading principle should be to use only such reagents, and a minimum quantity of them, as would introduce the least possible risk in the analysis by introducing the substance that was being sought for. In the case of a water containing lithium, just sufficient barium hydrate would be added to precipitate all the alkaline earth metals, with as little excess as possible; and the residue obtained, after elimination of the excess of barium, treated with a mixture of absolute alcohol and ether. On evaporation an residue was obtained, frequently deliquescent after exposure, which showed the characteristic lithium bands very distinctly, when such bands could not be seen by direct spectroscopic examination of the solid residue.

Mr. BLOUNT said that he could quite confirm Mr. Fairley's remarks in reference to lithia. Lithia was a very common constituent of natural water, in almost any sample of which he would undertake to find it by the method described by Mr. Fairley.

Mr. CHAPMAN said that in the determination of alkaline carbonates by evaporating to dryness and extracting with water it was necessary, in the case of waters containing appreciable quantities of magnesium salts, to take care that the washing was not carried too far, or the results obtained would be too high, owing to the distinct solubility of magnesium carbonate. With regard to the suggestion that carbonate of soda could not coexist in water with lime salts, if bicarbonate of soda solution were added to water containing several grains per gallon of sulphate of lime or sulphate of magnesia, the water remained perfectly bright. The conclusion he had come to was that those who systematically made complete mineral analyses of water observed a good many facts not commonly stated in the text-books. He preferred to express the results of such analyses, not in terms of the water, but in terms of the residue. He would like to ask Mr. Fisher whether he had formed any hypothesis to explain the presence of what was an extremely common constituent of pure waters of the class referred to, namely, saline ammonia in quantities, which in any other waters would be held to indicate pretty extensive pollution. He was not quite at one with Mr. Fisher in thinking that these waters rarely contained any appreciable quantity of lime or magnesia. In his experience, waters from similar beds in other parts of the country, while they contained often not more than 2 or 3 grains per gallon of the carbonates of lime and magnesia, sometimes contained as much as 8 or 10 grains per gallon (but not often more) of calcium carbonate.

Mr. C. A. MITCHELL inquired at what temperature the total solids were dried. It was rather difficult to expel all moisture at the ordinary temperature of the water-bath, while at a higher temperature magnesium chloride would be decomposed.

Mr. FISHER said that the total solids were dried on the water-bath.

The PRESIDENT (Dr. Voelcker) said that, to sum up Mr. Fisher's conclusions, they were to the effect that, in the case of the bare chalk, the alkaline carbonates were practically washed out entirely; but in the case of the chalk being covered by clay there was not such a thorough washing out, carbonate of soda being present to a very much larger extent than carbonate of lime, the carbonate of soda being originally an integral constituent of the chalk. This matter, of course, had to be looked at from several points of view and in regard to many different kinds of water. Analysts had been in the habit of saying that waters drawn from the chalk, but which were found to contain only a little lime, were to be regarded as somewhat abnormal, the explanation usually suggested being that there was some infiltration from the sea, and that chemical changes took place which resulted in the formation of carbonate of soda. He had before him some analyses of waters which would very well compare with the water which Mr. Fisher had referred to, namely, those of two supplies from deep artesian wells, situated respectively at the Royal Hotel, near Blackfriars Bridge, and in Stamford Street, near Waterloo Station. These waters, which he had analysed at different times, contained 50 grains of total solids per gallon, only 1 or 2 grains of lime, about ½ grain of magnesia, and 18 to 20 grains of carbonate of soda, together with large amounts of sulphate of soda; these results would lead one to similar conclusions to those which Mr. Fisher had arrived at. But there were other cases which did not quite bear this out, and he agreed with Mr. Chapman that there was not sufficient evidence of the non-existence of alkaline carbonates and lime salts together. In the case of a water from Newington, London, containing 86 grains of total solids per gallon, there were over 15 grains of lime per gallon, with large quantities of alkaline carbonates and alkaline sulphates as well. In some districts of Yorkshire, near Malton, for example, there were limited areas where deposits of carbonate of soda occurred, and the waters from the chalk in those neighbourhoods, although there was no overlying clay, frequently contained as much as 150 grains per gallon of carbonate of soda. He had made inquiries in the neighbourhood, and of geological authorities, but had never been able to find any exact records of the occurrence of these deposits or any satisfactory explanation of their presence.

Mr. Dibdin thought that probably biology would afford some clue to the means by which the carbonate of soda was produced. When the chalk was deposited it originally contained a certain quantity of organic matter derived from marine organisms, and it seemed quite possible that, as the result of the microbic action that would take place during the infiltration of an aerated water through the chalk, the carbon of this organic matter had become converted into carbonic acid, and under these bacterial conditions it might have been that some of the original sulphate of soda underwent change, resulting in the formation of sulphate of lime and carbonate of soda.

Mr. Fisher said that he had been thinking over this matter for years, and had rejected various explanations, until he obtained the direct evidence of the presence of carbonate of soda in the chalk, which seemed to decide the question. He had come to the conclusion that such was the probable explanation before he was able to obtain practical evidence of it. He hoped to be able to get evidence that the limestones in the other beds also contained free alkaline carbonates, which he thought would add to the strength of the position. What in the tables he had termed "alkalinity" represented all the carbonates as carbonate of soda. The figures give some information as to the relative alkalinity of the samples. A hundred c.c. of ordinary chalk-water generally neutralized about 5 c.c. of decinormal acid, but some of these samples neutralized 7, 8, 9 or 10 c.c., which afforded an idea of the amounts of alkaline material present, translated from decinormal acid to carbonate of soda. He was quite familiar with the district referred to by Mr. Richmond. The gault clay which occurred in the neighbourhood, and which varied in width from two to four miles, was a great difficulty in the way of getting proper water. A supply by gravitation was difficult owing to the distance from the outcrop of the chalk and the greensand, while if the clay were pierced the water obtained was extremely saline. The water from a deep well at Swindon was so saline that as it came down the Thames, at times, it affected the chlorine in the Oxford water-supply. This saline water had been traced by him in the Thames up to Lechlade, and from thence to Swindon by

Mr. Groves. On the 1 inch to the mile geological Ordnance map it would be seen that there was good reason for the variations in the quality of the water coming from below the gault. In some places the collecting-ground was large, and consisted to a considerable extent of exposed beds of coral rag, and the water came down gradually beneath the hills. In other places the Oxford clay covered the porous collecting-ground, and there was only a very narrow strip of pervious rock which could get any water at all. Shrivenham Station was just on the edge of the gault, and a little further north the coral rag was exposed, and there no excess of saline matter was to be observed. If the explanation were correct as to the cause of the alkalinity, it ought to apply to any similar bed of chalk below the London clay. There was such a bed of London clay in the southern part of Hampshire, near the New Forest, on both sides of which the chalk downs rose to a considerable height, and he hoped to have an opportunity of examining a sample from this locality. All these saline waters contained exceptional quantities of ammonia, and some contained considerable quantities of organic matter. At Marsh Gibbon, where the water flowed out of a borehole, it came out quite brown, from the quantity of organic matter dissolved in passing under the ground for three miles; that water contained also a notable quantity of free ammonia. Where ammonia was present in these waters there was very seldom any quantity of nitrates. The question of the solubility of calcium carbonate in waters containing an excess of free ammonia was dependent to some extent upon the proportion of carbonic acid present. In a soda-water made from a hard water considerable quantities of carbonate of lime would be found in solution as well as carbonate of soda. It was generally the case that those waters which were alkaline contained very small proportions of carbonate of lime, though he was not certain as to how the lime might have disappeared. Frequently there was only so much lime in solution as would dissolve *per se* as calcium carbonate.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

Methods for the Detection and Estimation of Arsenic in Beer, Brewing Materials, and Food. W. Thomson and J. P. Shenton. (*Journ. Soc. Chem. Ind.*, 1901, xx., 204.)

Marsh-Berzelius Test.—Fifty c.c. of *beer* are placed in an 8-ounce flask, and heated to the boil with agitation, then evaporated to a syrup on a sand-bath; 20 c.c. of nitric acid and 5 c.c. of strong sulphuric acid are next added and the liquid boiled down till it darkens. One or 2 c.c. more of nitric acid are introduced as often as discoloration occurs until the organic matter is completely destroyed (about 25 c.c. are usually required). The liquid finally occupies a volume of about 5 c.c.; it is diluted with an equal volume of water and boiled, to expel nitrous and nitric acids.

It is then made up to 15 c.c. The Marsh apparatus consists of a 50 c.c. conical Jena flask fitted with a wooden stopper, made tight with paraffin wax. It is conveniently provided with a graduated stoppered tube-funnel, holding 15 c.c., to contain the arsenical liquid, the stop-cock of which is so arranged as either to permit introduction of measured quantities of the arsenical liquid, or of dilute sulphuric acid (1 : 7). The flask is filled one-third full of granulated zinc. The hydrogen passes through a tube containing a coil of filter-paper and a plug of cotton-wool, then enters a hard glass tube $\frac{3}{16}$ inch in diameter, drawn out at the end. The acid should be added so that the hydrogen flame is between $\frac{3}{32}$ and $\frac{1}{8}$ inch long; and the Bunsen burner is placed where it will heat $\frac{3}{4}$ inch of the tube to redness about $\frac{3}{16}$ inch from the constriction. Sufficient of the arsenical liquid is gradually introduced (1, 2, or 3 c.c.) to yield a mirror of standard size, and thus the process becomes quantitative. Standard mirrors are prepared by adding from weak standard arsenious acid solutions quantities corresponding to the $\frac{1}{500}$, $\frac{1}{400}$, $\frac{1}{300}$, $\frac{1}{200}$, $\frac{1}{100}$, $\frac{1}{75}$, $\frac{1}{50}$, and $\frac{1}{25}$ grain per gallon. In making the comparisons, the flask should be as small as possible, and all dimensions and weights, etc., should be kept identical. Pure sulphuric and hydrochloric acids are obtained by distillation with a little chromic acid, chlorine in the HCl being destroyed by pure phenol. Rod zinc should not be used, and the metal should be quite pure, since various impurities, not all of which are known, tend to hold back the arsenic. Additions of platinum, platinic chloride, aluminium, magnesium, and lead or zinc salts, should be avoided.

In the case of *malt*, 5 grammes are treated with 25 c.c. of nitric acid, heated cautiously, and 5 c.c. of sulphuric acid added, proceeding then as above. With *coal* and *coke*, 5 grammes of the former, or 2·5 grammes of the latter, in fine powder, are heated with 25 c.c. of nitric acid for one hour, then with 5 c.c. of sulphuric acid for half an hour. After cooling, the mixture is diluted to about 100 c.c. and filtered. The filtrate is concentrated with more nitric acid to destroy organic matter, and finally evaporated free from nitrous and nitric acid. The resulting solution is then examined for arsenic as above.

REINSCH TEST.—The present authors cannot accept the process specified by the Brewers' Association (this volume, p. 13): (1) Because it is less delicate; (2) because it is less distinct than the Marsh test, the colour of the sublimate obtained in the Reinsch test not being so convenient for comparison as the Marsh black; (3) because the whole of the arsenic is not recovered from the copper by heating; (4) because some arsenic will be volatilized when the beer is concentrated with hydrochloric acid; (5) because arsenic acid may be present and delay the reaction; and (6) because the small piece of copper is not likely to collect all the arsenic present in the liquid.

MARSH-GUTZEIT TEST.—This, as modified by Tyrer, was tested. The arsine was passed through potash bulbs charged with strong lead acetate solution before being applied to the paper treated with mercuric chloride. The gas does not appear to dissolve in the solution to any appreciable extent; and the authors consider this the best method of working the test, questioning whether the Marsh-Gutzeit is not the best for the approximate determination of minute quantities of arsenic after the organic matter of the original sample has been destroyed. One disadvantage is that the yellow colour cannot be seen by gaslight.

Neither sulphuric nor hydrochloric acid free from chlorine removes arsenic from arsenical glass when stored therein for seventeen hours in the cold. Sodium selenite yields a dark-brown precipitate in the Marsh flask, and gives a bright red mirror. No indication of that element was discovered in any of the beers and sugars examined.

F. H. L.

ORGANIC ANALYSIS.

A Gas-volumetric Method of Determining Formaldehyde. E. Riegler. (*Zeit. anal. Chem.*, 1901, xl., 92-94.)—If hydrazine sulphate be treated with a solution of iodic acid, the whole of its nitrogen is liberated, as in the equation:

$$5(N_2H_4H_2SO_4) + 4HIO_3 = 5N_2 + 12H_2O + 5H_2SO_4 + 4I.$$

If, however, formaldehyde be present, it combines with the hydrazine to form a hydrazone, which is only decomposed after some time by the iodic acid. Thus, a quantity of hydrazine corresponding to the hydrazone remains undecomposed, and the volume of nitrogen is smaller.

The reagents required are a solution of 1 gramme of hydrazine sulphate in 100 c.c. of water, and a solution of 5 grammes of crystallized iodic acid in 50 c.c. of water.

The apparatus used by the author was the well-known Knop-Wagner nitrometer. Twenty c.c. of the hydrazine solution are mixed with 20 c.c. of water in the evolution flask, and 5 c.c. of the iodic acid solution introduced into the inner vessel. The nitrogen is collected and measured in the usual manner.

A definite quantity of the aqueous solution of formaldehyde containing not more than 0·08 gramme of that substance is now shaken with 20 c.c. of the hydrazine sulphate solution, and the mixture left for fifteen minutes. It is then introduced into the evolution flask of the apparatus, together with about 10 c.c. of water, and the nitrogen liberated as before by the introduction of 5 c.c. of the iodic acid solution, the shaking being continued for half a minute, and the reading taken after two minutes (not more).

The difference between the two results, calculated to standard temperature and pressure, and multiplied by the factor 2·7, gives the corresponding amount of formaldehyde in milligrammes.

C. A. M.

The Separation and Determination of Ammonia and Methylamines. H. Quantin. (*Ann. de Chim. anal.*, 1901, vi., 125, 126.)—This method is based on the following principles: If a solution containing both free or combined ammonia and methylamines be treated with sodium phosphate and then with magnesium sulphate, the whole of the ammonia is precipitated as ammonium magnesium phosphate, provided the liquid be alkaline from the presence of methylamines, which should be added if necessary.

An aliquot part of the solution is diluted to a litre:

(*a*) The total volatile bases are determined in 100 c.c. of this solution, and calculated as ammonia = A.

(*b*) One hundred c.c. of the solution are treated with sufficient sodium phosphate and magnesium sulphate to precipitate a quantity of ammonia equal to A. Purified methylamines are added to insure the complete precipitation, and the precipitate allowed to settle for six hours, after which it is collected and washed with water until the washings are neutral.

(*c*) The precipitate and filter-paper are transferred to a distillation flask, the precipitate dissolved in hydrochloric acid, an excess of sodium hydroxide added, and the ammonia distilled into standard acid in the usual manner.

(*d*) One hundred c.c. of the original solution are concentrated to a small volume, the total ammonia and mono- and di-methylamines precipitated with platinum chloride, and the precipitate collected, dried, and weighed. Let p represent the weight of the precipitate, and p_1 the weight of platinum obtained on igniting the precipitate. Seventeen parts of ammonia, 31 parts of monomethylamine, and 45 of dimethylamine yield respectively 223, 237, and 251 parts of platinochlorides, containing 98·5 parts of platinum.

If the quantities of the three bases be represented by x, y, z, we obtain the equations:

$$(1)\quad \frac{223}{17}x + \frac{237}{31}y + \frac{251}{45}z = p;$$

and

$$(2)\quad \frac{x}{17} + \frac{y}{31} + \frac{z}{45} = \frac{p_1}{98{\cdot}5}.$$

Substituting for x the result from the determination of ammonia, two equations are obtained, giving the values of y and z.

Finally, the difference $A-(x+y+z)=P$ gives in terms of ammonia the proportional amount of trimethylamine, of which the real quantity will be $\frac{P\times 59}{17}$.

[Ammonium magnesium phosphate does not readily yield its ammonia on mere distillation with sodium hydroxide.—B. D.]

C. A. M.

Quantitative Determination of Malic Acid. A. Hilger. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1901, iv., 49, 50.)—The author proposes a method based upon the observed fact that 1 gramme of malic acid, on boiling with a slightly alkaline or neutral solution of palladium chloride reduces 0·294 gramme of palladium. One hundred c.c. of the liquid—*e.g.*, wine—under examination are evaporated, to remove volatile constituents, and the residual liquid made feebly alkaline with basic lead acetate. The resulting precipitate, which contains the whole of the malic acid, is filtered off, washed with cold water, and dissolved in a little dilute boiling acetic or nitric acid. Sodium carbonate is then added to the boiling solution to alkaline reaction, and a current of carbonic acid gas passed through the liquid. The precipitate of basic lead carbonate is removed by filtration, the filtrate concentrated to 100 c.c. It is then neutralized with hydrochloric acid, transferred to a 500 c.c. Erlenmeyer flask, treated with 10 c.c. of a 5 per cent. palladium chloride solution, and boiled for ten minutes. The reduction of the palladium chloride is accompanied by a brisk disengagement of carbonic acid gas; when this ceases, the liquid is made faintly acid with hydrochloric acid, and the heating continued until the palladium has consolidated and settled to the bottom of the vessel. The precipitated metal is then filtered off, washed, dried, heated in a stream of carbonic acid gas, cooled, and weighed. H. H. B. S.

Remarks upon the Halphen Reaction with Cottonseed Oil. E. Wrampelmeyer. (*Zeit. für Untersuch. der Nahr und Genussmittel*, 1901, iv., 25.)—The author considers that Soltsien's modification of Halphen's procedure (*Zeit. öffent. Chem.*, 1899, 106, 135) impairs the sensitiveness of the test. According to his experience, a decided indication can be obtained in a quarter of an hour by the use of amyl alcohol, as stated by Halphen, whereas by Soltsien's procedure three-quarters of an hour are required. He, however, does not consider it necessary to revert to the use of the salt-water bath. The author's method of working is as follows: A moderately thick test-tube, about 2·5 centimetres wide and 15 centimetres long, and graduated at 10 centimetres, is filled to the graduation with the oil to be examined, and an equal volume of amyl alcohol and about 2 c.c. of a 1 per cent. solution of sulphur in carbon disulphide added. A bored cork with fitted tube is then inserted in the neck of the test-tube, and the whole heated in a boiling-water bath for a quarter of an hour (*cf.* ANALYST, 1898, 131; 1899, 214; 1900, 105). H. H. B. S.

The Optical Examination of Fats and Waxes. G. Marpmann. (*Chem. Rev. Fett. u. Harz. Ind.*, 1901, viii., 65-68.)—Although the refractive index of beeswax affords a rapid means of judging as to its impurity, there has hitherto been the drawback that the refractometer is injured by being brought to the necessary temperature of 70°-80° C.

This can be obviated, however, by making use of the fact that the refractive index of a mixture is equal to the arithmetical mean of the indices of its components. Thus, by mixing beeswax with a fatty oil, a mass which remains fluid at 40° C. can be obtained, and its refraction index readily determined.

Ethereal oils are also more or less suitable substances for the admixture, but ether, chloroform, or higher alcohols, benzene, etc., are less satisfactory. The author gives the results of experimental tests with mixtures of beeswax with peppermint oil and clove oil. C. A. M.

Determination of Cresol. F. Russig and G. Fortmann. (*Zeits. angew. Chem.*, 1901, 157.)—The following method gives somewhat higher results than that of Raschig (ANALYST, 1900, 298). 50 grammes of the cresol are weighed into an Erlenmeyer flask, and 125 grammes of sulphuric acid of 66° B. are added. The mixture is warmed to 60° or 70° C., and allowed to stand one or two hours. The nitrating apparatus consists of a glass retort of about 1 litre capacity fitted with a dropping funnel. Into the retort 400 grammes of nitric acid of 40° B. are introduced and heated to 60° C., the flame being then removed. The sulphuric acid is allowed to drop into the nitric acid. This takes one and a half to two hours. About twenty minutes after all has run in, the contents of the retort are poured into a large porcelain basin containing 200 c.c. of water, and a further 200 c.c. of water are used to wash out the retort. It is then allowed to stand overnight. The trinitro-cresol is then collected on a hardened filter-paper, washed with 200 c.c. more water, dried at 95° to 100°, and weighed. Pure metacresol thus yields 175·6 per cent. of trinitro-cresol as against 174 per cent. given by Raschig's method.

The authors find that the method of Ditz and Cedivoda (*Zeits. angew. Chem.*, 1900, 1050) is quite unreliable. A. M.

Determination of Cresol. H. Ditz. (*Zeits. angew. Chem.*, 1901, 160.)—The author replies to the criticisms of Russig and Fortmann upon his method (see above). A. M.

INORGANIC ANALYSIS.

The Estimation of Manganese in Ferro-Chromium Alloys. J. T. (*Chem. News*, vol. lxxxiii., p. 25.)—One gramme of the powdered alloy is fused with several grammes of sodium peroxide in a nickel crucible, then dissolved in water, boiled for five to ten minutes, and filtered. The mixed oxides of iron, manganese and nickel filter badly, but settle readily, and should therefore be boiled repeatedly with very dilute ammonium carbonate, and collected on the filter when the liquid has nearly or quite lost its yellow colour. The residue and (asbestos) filter are treated with equal parts of nitric acid (1·20) and water, and sufficient ferrous sulphate to fully reduce the nickel and manganese oxides. The solution is thoroughly cooled, oxidized with sodium bismuthate, and the filtered permanganate titrated with hydrogen peroxide, according to the instructions of Reddrop and Ramage.

The green colour of the titrated solution, due to nickel from the crucible, is destroyed by a slight excess of permanganate. C. S.

On Vogel's Method for the Qualitative Detection of Cobalt. F. P. Treadwell. (*Zeit. anorg. Chem.*, xxvi., 108.)—This method is especially suitable for the detection of traces of cobalt in nickel salts, even in the presence of iron. To the solution of the nickel salt a concentrated solution of ammonium thiocyanate is added, and the whole shaken with a few c.c. of amyl alcohol. If the upper alcoholic layer remains colourless, neither iron nor cobalt is present; if it becomes red, iron is indicated. In this case a few drops of sodium hydroxide solution are added, and the liquid is again shaken; if cobalt is present, the upper layer will now be coloured blue, through the formation of a compound $Co(NH_4)_2(CNS)_4$. A. G. L.

Estimation of Aluminium in Steel. E. Spatz. (*Zeits. öffentl. Chem.*, 1901, vii., 60; through *Chem. Zeit. Rep.*, 1901, 79.)—One gramme of filings is dissolved in 2 c.c. of strong sulphuric acid and 15 or 20 c.c. of water. The solution is transferred to a silver basin, treated with 40 grammes of crystallized ammonium oxalate, and electrolyzed. [No electrical measurements are given.] At intervals a few drops of the liquid are withdrawn through a fine pipette, evaporated and ignited on platinum foil, and tested for iron. When only traces thereof remain, the main solution is run into another vessel, and the former basin is rinsed out and rubbed well with a rubber-tipped glass rod. The liquid is evaporated to dryness, brought to a dull red heat, and fused with potassium bisulphate. The melt is dissolved in water, acidulated with sulphuric acid, mixed with some ammonium phosphate, and precipitated with ammonia; the precipitate is collected, dissolved in hydrochloric acid, thrown down again with ammonia, filtered, dried, and weighed as iron plus aluminium phosphate. It is then once more fused with bisulphate, the melt taken up in dilute sulphuric acid, a weighed piece of pure zinc added, and the iron titrated with permanganate. By calculating the weight of iron found into phosphate, and deducting from the mixed phosphates, the amount of aluminium phosphate derived from the steel is left. If the sample only contains traces of aluminium, 4 or 5 single grammes should be electrolyzed separately, and the united liquids examined as above.

F. H. L.

Calcium Compounds in Soil. Estimation of "Available" Lime. D. Meyer. (*Landw. Jahrb.*, 1900, xxix., 913; through *Chem. Zeit. Rep.*, 1900, 377.)—The author describes an investigation of twenty-six different specimens of soil, on twenty-one of which experiments with growing plants were also carried out. The total lime in the samples varied from 0·092 to 1·271 per cent. The average proportion of lime in the light soils was 0·333 per cent., in the heavy soils 0·694 per cent.; but although the latter usually contained more than the former, this was not always the case. In twenty-two of the samples the proportion of carbon dioxide ranged from 0·020 to 0·076 (mean 0·045) per cent.; four samples contained from 0·168 to 0·350 per cent. of CO_2. The average for the light soils was 0·052 per cent., for the heavy soils 0·098 per cent. Most of the soils contained less than 25 per cent. of their total lime as carbonate; there was a larger proportion of carbonates in the light than in the heavy soils. Samples having the same proportion of carbon dioxide contained totally different proportions of lime.

The solubility of the lime in dilute hydrochloric acid varied considerably; in light soils it was less soluble than in the heavy soils. There was no connection between the solubility of the lime in dilute hydrochloric acid and the proportion of very fine particles in the soil. There was no connection between the amount of carbon dioxide, of sulphuric acid, or of phosphoric acid and the solubility of the lime. Magnesia stood in much the same relation as the lime. Judging from the small proportion of carbon dioxide, sulphuric acid, and phosphoric acid, it is clear that most of the lime in these soils existed as silicates. Since the solubility of the lime in dilute acid was less in the light than in the heavy soils, the latter must contain their lime chiefly as easily decomposable silicates.

Different compounds of lime exhibit notable differences in their effects upon the growth of plants. The useful compounds of calcium are, according to the author, the carbonate, the sulphate, and the easily decomposable silicates; and as they can be dissolved by a neutral solution of ammonium chloride or nitrate, by adopting these solvents it becomes possible to devise a process for estimating available lime which gives figures agreeing better with the results of experiments on plants than when hydrochloric acid is employed.

Meyer accordingly proposes to estimate the "available" lime by digesting the sample of soil for three hours on the water-bath with a 10 per cent. solution of ammonium chloride, determining the calcium in the filtrate without removing the silica. A yield of 0·25 per cent. by this process may be considered normal; it should not fall below 0·20 per cent. F. H. L.

On the Quantitative Determination of Magnesium by Means of Organic Bases. W. Herz and K. Drucker. (*Zeit. anorg. Chem.*, xxvi., 347.)—Magnesium can be separated from the alkali metals in the same way as zinc, by precipitating the solution in the cold with an aqueous solution of dimethylamine; after standing for several hours the precipitated magnesium hydroxide is filtered off, washed rapidly with water containing dimethylamine, and ignited over a Teclu burner. The results obtained were satisfactory. The separation may also be effected by means of guanidine, obtained from the carbonate by adding an amount of barium hydroxide insufficient to liberate all the guanidine. The carbonate itself does not precipitate magnesium completely. A. G. L.

Determination of Fluorine in Zinc Blendes. F. Bullnheimer. (*Zeits. angew. Chem.*, 1901, 101.)—The apparatus used consists of a flask of about 300 c.c. capacity fitted with a rubber stopper, through which pass inlet and outlet tubes and a thermometer. The outlet tube leads first through a U-tube filled with glass wool, then through a cooled Winkler coil into a Drehschmidt wash bottle containing potassium chloride solution. About 2·5 grammes of the blende are weighed out, mixed with 3 to 5 grammes of quartz powder, and introduced into the flask. A mixture of 20 grammes of chromic acid and 100 c.c. of concentrated sulphuric acid are added, the flask is closed, shaken round, and purified air is led through the apparatus. After a time the temperature of the contents is gradually raised to 80° C. An energetic reaction then sets in, and the flame is removed. When this has ceased, the temperature is raised to 150°, and the heating is continued for three hours. The whole of the fluorine will then have been driven off, and will have liberated acid according to the equations:

$$3SiF_4 + 2H_2O = 2H_2SiF_6 + SiO_2$$
$$2H_2SiF_6 + 4KCl = 2K_2SiF_6 + 4HCl.$$

The acid is titrated with decinormal alkali, using phenolphthalein as indicator. A. M.

A New Test for Chlorine for Use with the Blow-pipe. Henry W. Nichols. (*Amer. Chem. Journ.*, xxv., 315.)—The substance to be examined is heated with potassium bisulphate in a tube closed at one end and a piece of filter-paper moistened with a solution of cobalt nitrate held at the mouth of the tube. If a chloride is present, the paper will turn blue; if a bromide or iodide, green. In the case of small quantities of chlorides, it may be necessary to dry and warm the paper to produce the colour. If both chlorides and bromides are present, the paper turns blue first, and then green, or, if but little bromine is present, bluish-green. A mineral containing about 2·2 per cent. of chlorine gave a good reaction by this method, but the test is much less sensitive for bromides than for chlorides.

Chlorides in solution may be detected by dipping a piece of filter-paper into the liquid, adding a drop of cobalt nitrate, and drying the paper, but the test is not very delicate.

The chlorochromic acid test may be modified by carrying it out in a plain test-tube, and holding a piece of filter-paper moistened with a saturated solution of sodium carbonate at the mouth of the tube. The paper retains the chromium, which can be easily identified in the ash. A. G. L.

Proximate Analysis of Clays. W. Jackson and E. M. Rich. (*Journ. Soc. Chem. Ind.*, 1900, xii., 1087.)—In order to estimate the amount of clay substance, felspar, and quartz in a sample of clay, it is usual to utilize the action of sulphuric acid on hydrated silicate of alumina (clay), which brings about the solution of the alumina as sulphate. The silica remains in the amorphous condition, and is subsequently taken up with dilute alkali. The authors find, however, that both acid and alkali act upon felspar, dissolving it as a whole, especially in the case of plastic clays. A. M.

On the Persulphates and their Application in the Laboratory and Industries. R. Namias. (*L'Orosi*, xxiii., 218, through *Zeit. anorg. Chem.* xxvi., 125.)—For the analysis of persulphates, the salt should be allowed to stand in the cold for twelve hours with potassium iodide solution, and the liberated iodine titrated. As solutions of persulphates remain unchanged for a long time, they can be used in volumetric analysis. Ammonium persulphate precipitates manganese dioxide quantitatively from hot ammoniacal solutions of manganese salts. When used in excess in hot solutions, it is also capable of oxidizing chromic oxide to chromic acid in one hour. A. G. L.

The Recalculation of the Chemical Analyses of Rocks. J. F. Kemp. (*School of Mines Quart.*, xxii., 75.)—If the chemical analysis of a rock is accompanied by a microscopical study of thin sections of the rock, it is often possible to calculate the percentages of the several minerals present. In the first place, the "molecular ratios" of the various molecules present are determined by dividing the percentage values found by the corresponding molecular weights. The following example of a syenite is taken from a report by W. H. Weed and L. V. Pirsson (Twentieth Annual Report Directory United States Geological Survey, iii., 466), the molecular proportions being given under the respective percentage values:

SiO_2.	Al_2O_3.	Fe_2O_3.	FeO.	MgO.	CaO.	Na_2O.	K_2O.	P_2O_5.	Cl.	Total.
64·64	16·27	2·42	1·58	1·27	2·65	4·39	4·98	0·37	0·05	98·62
1·077	0·158	0·015	0·022	0·031	0·047	0·070	0·053	0·002	0·0014	

The remaining 1·50 per cent. consisted of TiO_2, H_2O, CO_2, BaO, SrO, and is neglected in the recalculation. An examination of thin sections with the microscope showed the minerals present in the rock to be the following: Orthoclase, K_2O, Al_2O_3,

$6SiO_2$; plagioclase, albite Na_2O, Al_2O_3, $6SiO_2$, and anorthite CaO, Al_2O_3, $2SiO_2$; hornblende, MgO, SiO_2, CaO, SiO_2 and FeO, SiO_2; magnetite, Fe_2O_3, FeO; apatite, $9CaO$, $CaCl_2$, $3P_2O_5$; quartz, SiO_2.

On inspecting the above formulæ, it is evident that all the K_2O (53) is in the orthoclase, all the Na_2O (70) in the albite, and the remaining Al_2O_3 (35) in the anorthite, requiring an equal number of molecules of CaO (35). The rest of the CaO (47) is in the hornblende and apatite, and the apatite can be calculated from the P_2O_5 (2). All the Cl_2 (·7) is in the apatite, and the MgO (31) in the hornblende. All the Fe_2O_3 (15), and an equivalent amount of FeO (15), are in the magnetite, the remaining FeO (7) being in the hornblende. The excess of SiO_2 constitutes the quartz.

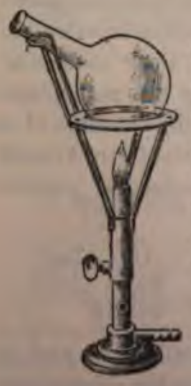
To obtain the percentages of the minerals in the rock, the molecular proportion of one of the constituents of the mineral is multiplied by the molecular weight of the mineral—*e.g.*, the percentage of orthoclase is $0·053 \times 556 = 29·46$. Thus the following percentages are obtained: Magnetite 3·50, hornblende 4·60, anorthite 9·73, albite 36·68, orthoclase 29·46, quartz 13·54, apatite 1·10: Total, 98·61.

The ratio of the albite molecules to those of anorthite is as 4 to 1. From the optical properties of the plagioclase, Pirsson found it to be approximately Ab_2An. Consequently half the albite is in the orthoclase, and the ratio of alkali felspar to soda-lime felspar is as 424 Or + 280 Ab to 280 Ab + 140 An, or nearly as 5 to 3. By means of a result of this nature, a rock may be readily classified as an orthoclase or plagioclase rock.

In some cases, however, the recalculation is not so simple as in the above example, and it may become necessary to separate and analyse one of the minerals before proceeding with it. The author gives the formulæ of the more important minerals, and also a set of tables giving the molecular ratios corresponding to different percentages of various oxides.

A. G. L.

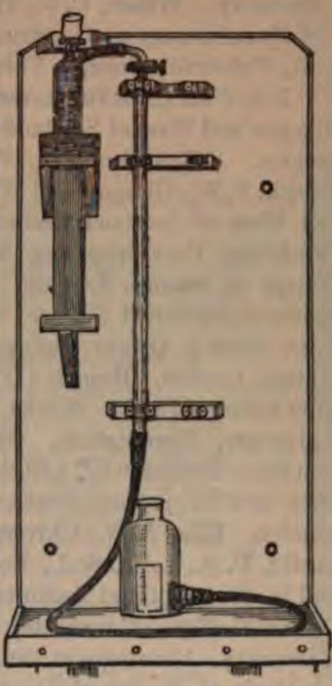
APPARATUS.



A Flask Support. **F. Reiss.** (*Chem. Zeit.*, 1901, xxv., 351.)—This device consists of a round-topped tripod carried on a short sleeve, which fits over the tube of a Bunsen burner, and which bears a thumbscrew for clamping it at the desired height. The upper ring also supports a suitably bent wire to hold the neck of a flask whilst being heated in a sloping position, as is necessary in working the Kjeldahl process (for which the stand is primarily intended)—Obviously the neck support can be omitted and the sleeve increased in length indefinitely; and thus the apparatus forms the only stand required to hold a flask or retort before the mouth of a condenser the gas flame burning at the upper orifice of the sleeve itself.

F. H. L.

A Form of Volumenometer. F. W. Jones. (*Chem. News*, vol. lxxxiii., p. 100.)—The apparatus is designed for specific gravity determinations in the case of substances which cannot be brought into contact with water, and consists of a gas burette with stopcock, connected at one end with a stoppered bottle, and at the other end with a reservoir of mercury. The bottle should be small, and have a stopcock fitted to one of the two openings in the ground stopper. It is mounted on a spring platform, whilst the stopper is fixed, and the second opening is connected, by a rubber tube, with the burette, which, in turn, is connected to the mercury reservoir as usual.



A known weight of substance being placed in the bottle and the stopcocks opened, the reservoir is raised until the mercury just reaches the top of the burette; whereupon the burette stopcock is closed and the bottle placed on the lower shelf, the barometric pressure of the mercury being then read off. When the air in the bottle has attained room temperature, the bottle stopcock is closed and that of the burette opened, the fall of the mercury in the burette and its rise in the reservoir being noted.

The results are calculated by the formula: $V_1 = v\frac{d+r}{P-d-r}$ wherein V_1 is the gas volume of the bottle and connections; P = the pressure; v the volume of gas in the burette at the reduced pressure; d the amount the mercury falls in the burette; and r the amount of its rise in the reservoir. Then, V being the volume of bottle and connections when empty, the specific gravity is $\frac{W}{V-V_1}$.

C. S.

INSTITUTE OF CHEMISTRY OF GREAT BRITAIN AND IRELAND.

The following is a list of the names of the candidates who passed the examination of the Institute of Chemistry held in July, 1901:

Intermediate Examination.—Armstrong, A. G., Glasgow and West of Scotland Technical College, and at King's College, London. Arnaud, F. W. F., King's College, London, also under Messrs. W. F. Lowe and C. H. Cribb, FF.I.C. Brooker, J. W. G., Finsbury Technical College, London. Finnemore, H., Pharmaceutical Society's Laboratory, and at King's College, London. Green, G. F. D., King's College, London, and at Central Technical College, London. Jones, E. V., University, Birmingham. Kirkhope, T. B., Glasgow and West of Scotland Technical College. Munro, W. T., Glasgow and West of Scotland Technical College. Neville, H. A. D., B.Sc. (Lond.).

Peters, W. H., University College, Nottingham. Walker, J. H., Glasgow and West of Scotland Technical College. Wallis, T. E., B.Sc. (Lond.), Pharmaceutical Society's Laboratory. Wilson, C. J., Finsbury Technical College, London.

Examination in General Practical Chemistry for Fellowship.—Coppock, J. B., University College, Nottingham, and at Royal College of Science, London.

Final A.I.C. Examination.—Branch "A" (*Mineral Chemistry*): McLellan, B. G., Glasgow and West of Scotland Technical College. Northall-Laurie, D., King's College, London. O'Shaughnessey, F. R., A.R.C.Sc., Royal College of Science, London. Tainsh, P. W., Glasgow and West of Scotland Technical College. Taylor, T., Glasgow and West of Scotland Technical College. Scott Tebb, W., M.A., M.D. (Cantab.), Cambridge University and King's College, London. Wade, F., A.R.C.Sc., Royal College of Science, London. Watson, F. W., B.Sc. (Lond.), Glasgow and West of Scotland Technical College. Branch "B" (*Metallurgical Chemistry*): Abell, C. T., B.Sc. (Vict.), Owens College, Manchester. Comber, A. W., Finsbury Technical College, London. Branch "D" (*Organic Chemistry*): Dakin, H. D., B.Sc. (Vict.), Yorkshire College, Leeds. Hinks, E., King's College, London. Slator, A., B.Sc. (Lond.), University, Birmingham. Smith, R. E. B., B.Sc. (Lond.), University College, London. Branch "E" (*Analysis of Food and Drugs and of Water, including Examination in Therapeutics, Pharmacology and Microscopy*): Coysh, B. R., King's College, London. Ellis, A. W., University, Birmingham, and under J. K. Colwell, Esq., F.I.C. Jardin, D. S., A.R.C.Sc.I., Royal College of Science, Dublin. Macara, T., Glasgow and West of Scotland Technical College, and under Dr. J. Clark, F.I.C. Melling, S. E., Owens College, Manchester, and under A. H. Allen, Esq., F.I.C. Webster, J., University, Birmingham, and under J. K. Colwell, Esq., F.I.C. Branch "E" (*Analysis of Food and Drugs and of Water*): Brown, J. A., University College, Nottingham.

The examiners in Chemistry were Dr. Bernard Dyer, F.I.C., and Dr. W. Palmer Wynne, F.R.S., F.I.C. The examiner in Therapeutics, Pharmacology and Microscopy was Dr. A. P. Luff, F.R.C.P., F.I.C.

ARSENIC COMMITTEE.

The Joint Committee of the Societies of Chemical Industry and Public Analysts have circulated a number of samples containing definite quantities of arsenic, and have carefully considered the results returned by the members. The Committee believe that these results justify them in prescribing a method for dealing with the various substances, but before taking this course they think it desirable to submit this method to somewhat more extended trials.

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THE ANALYST.

SEPTEMBER, 1901.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

NOTE ON THE REDUCTION OF FERRIC SALTS.

By Gilbert T. Morgan, D.Sc.

(*Read at the Meeting, May* 1, 1901.)

Attention has often been drawn to the fact that zinc is far from being an ideal reducing agent for ferric salts. Scheiding (*Zeit. angew. Chem.*, 1895, 79) has shown that very elaborate precautions, involving the use of a large excess of zinc and very dilute iron solutions, are necessary in order to obtain trustworthy results, one important point being the digestion of the materials in the cold for five or six hours. Even after dissolving a considerable excess of the metal in boiling acidified solutions of ferric salts, it is frequently noticed that an appreciable amount of iron still remains in the higher state of oxidation, the hydrogen evolved from zinc dissolving in cold solutions seems to be more efficacious in reducing ferric iron than that set free at higher temperatures; but, on the other hand, the dissolution of the metal proceeds somewhat slowly in the cold, and hence the method is not suitable for practical requirements. Various modifications of the process have been put forward with a view of accelerating the reduction. Beche (*Chem. News*, liii., 269) suggests the use of amalgamated zinc supported on platinum foil; Carnegie (*J.C.S.*, 1888, liii., 468) employs zinc-dust and feebly-acid solutions of the ferric salt: the reduction is very rapid, and the method gives accurate results when the reducing agent is free from iron and other substances which decolorize permanganate or dichromate solutions. In the following process, which admits of the use of pure zinc, either granulated or in sheets, the efficiency of the metal is considerably increased by employing it in the form of the zinc-copper couple.

A weighed portion (4 to 6 grammes) of ferric ammonium sulphate was dissolved in 100 c.c. of dilute sulphuric acid (3 per cent.) and the cold solution poured on to a zinc copper couple, produced by immersing 8 grammes of granulated zinc for two to three minutes in 200 c.c. of a 10 per cent. solution of copper sulphate; a vigorous action set in, and after ten minutes a drop of the solution no longer gave the thiocyanate coloration. The reduced solution, decanted from the couple and rapidly filtered to remove any floating particles of metal, was made up to 250 c.c. with recently-boiled dilute sulphuric acid, and aliquot portions were then withdrawn and titrated with standard permanganate solution.

The percentage of iron in the specimen of ferric alum employed was 11·50, as indicated by two concordant gravimetric analyses, whilst two volumetric estimations carried out in the manner indicated gave 11·34 and 11·44.

Under these experimental conditions the amount of zinc required to reduce the quantities of ferric salt usually taken for volumetric estimations is comparatively small, and the couple may be employed for several determinations. Owing to this diminution in the quantity of the zinc involved, the deduction to be made for the reducing action of the impurities in the metal becomes so small as to be almost negligible. In the foregoing experiments, where fairly pure zinc was employed, a blank experiment showed that less than 0·05 c.c. of the permanganate solution (1 c.c. = 0·0056 Fe) was decolorized, therefore for ordinary purposes no correction need be made.

In describing the zinc method of reduction, Fresenius (*Quanti. Chem. Analysis*, i., 224) emphasizes the importance of dissolving all the metal before titrating by referring to a statement made by Mitscherlich (*Journ. Prakt. Chem.*, 1861, lxxxvi., 3), to the effect that the zinc precipitates notable quantities of iron, which only redissolve as the reducing-agent passes into solution. At the time the observation was published, however, Fresenius seems to have been somewhat sceptical as to its accuracy (*Zeit. Analyt. Chim.*, ii., 72).

The foregoing results show that the precipitation of iron does not occur to any appreciable extent when the ferric salt is reduced by the zinc-copper couple, nor could any loss of iron be detected when the experiments were repeated with excess of granulated zinc, the ferrous solutions being decanted from the undissolved metal. Two concordant analyses made in this manner gave the percentage of iron in the alum as 11·47.

Further confirmation of these results was obtained by estimating the iron in pianoforte-wire. Weighed portions (0·3 to 0·5 gramme) dissolved in 30 c.c. of 15 per cent. sulphuric acid, oxidized to the ferric condition by potassium permanganate, and then again reduced to the ferrous state by the zinc-copper couple, were titrated with standard permanganate solution, two determinations giving the percentage of iron as 99·57 and 99·53.

Mitscherlich stated that the quantity of iron precipitated by the zinc is "ziemlich bedeutend," whereas these experiments indicate that, even if any such precipitation occurs, the consequent loss of iron is well within the limits of the experimental error of an ordinary volumetric analysis, and accordingly the ferrous solution may be separated from the reducing-agent as soon as the reduction of the ferric salt is complete.

The zinc-copper couple may be employed with advantage for reducing ferric salts in hydrochloric acid solution, the action being far more rapid than that of zinc or ammonium hydrogen sulphite; it is therefore applicable for the volumetric analysis of iron ores with standard bichromate solutions. The percentage of iron in a sample of red hematite was 64·13, as determined by the use of the couple, the value 64·04 being obtained by the ammonium hydrogen sulphite method.

When the zinc-copper couple or granulated zinc is added to a neutral solution of ferric alum a portion of the iron is precipitated as basic sulphate. The whole of the iron thus rendered insoluble is in the ferric condition, whereas the portion remaining dissolved is entirely converted by the reducing agent into the ferrous state. The brown precipitate, which rapidly forms when the couple is employed, readily dissolves in dilute acid, and the reduction then follows the normal course.

THE EXAMINATION OF COMMERCIAL MALT EXTRACTS.

By W. J. Sykes, M.D., and C. A. Mitchell, B.A.

(*Read at the Meeting, December* 5, 1900.)

Having had occasion to examine several samples of commercial malt extract, it was deemed advisable to extend the analyses so as to include typical preparations in the market. The methods employed and the results obtained are given below.

Such malt extract should be prepared from low-dried and therefore highly diastatic malts, so that the product may contain a large amount of diastatic power. In its manufacture, as a rule, the malt is first mashed in cold water, sufficient water at a temperature of 30° C. (186° F.) being subsequently added to bring the temperature of the mash to 55° C. (131° F.). The resulting wort is then evaporated under diminished pressure at a temperature not exceeding 55° C. (131° F.) until it has attained a syrupy consistence.

Malt with a diastatic power of 50 on Lintner's scale is readily obtained, and by special treatment malt with a much higher diastatic power than this can be manufactured.

In order to ascertain if it were possible to retain the whole of the diastatic power of the malt in the resulting extract the following experiments were made:

1. Wort from a malt with a diastatic power of 16·6 was evaporated very slowly under diminished pressure, CO_2 being passed through the flask when evaporation was not proceeding. Final product dark and turbid, diastatic power nil.

2. Wort from malt with a diastatic power of 18 concentrated in partial vacuum to one-fifth its bulk. Product turbid, and did not yield clear solution; evidently there had been some bacterial fermentation proceeding. Diastatic power 11.

3. Similar wort concentrated on water-bath at 100° to 110° F. and completed *in vacuo* over sulphuric acid. On diluting the product to the initial volume, a bright solution was obtained with a diastatic capacity of 18—that is, the same as the original.

From these it appears that it is possible to obtain a malt extract in the manufacture of which none of the diastatic power of the original malt has been lost.

Nitrogenous Constituents, Ash and Phosphoric Acid.—The amount of the soluble constituents of malt varies, it being dependent on these actually present in the raw barley, the temperature at which the malt is grown, and upon other conditions.

The natural variation in barley is well shown by the following results obtained by Gordon Salamon (*Journ. Soc. Chem. Ind.*, 1885).

PERCENTAGE OF SIXTEEN SAMPLES.

	Ash.	Total N.	Soluble N.	P_2O_5.
Highest	3·41	1·70	1·193	1·10
Lowest	2·09	1·27	0·662	0·635

The following are results obtained by ourselves from twelve different malts dried at temperatures of about 130° F. In each case a wort of a specific gravity of 1030 (water = 1000) was prepared at a temperature not exceeding 156° F., and the results multiplied by 10, so as to correspond approximately with an average commercial malt extract:

	Ash.	P_2O_5.	Total N.	N precipitated by $ZnSO_4$.	N as Ammonia.
Highest ...	1·66	0·72	0·69	0·09	0·14
Lowest	1·44	0·40	0·40	0·08	0·05
Average... ...	1·57	0·56	0·59	0·08	0·05

In reference to the influence of the temperature at which the malt has been germinated on the nitrogenous constituents, the following figures published by Day (1891) are instructive:

TEMPERATURE OF GERMINATION.

N.—substances soluble at 104° F. × 6·33 ...	40° F.	50° F.	55° F.	60° F.	65° F.	75° F.
Permanently soluble ...	2·70	4·13	4·19	3·80	3·49	3·29
Coagulated on boiling...	0·31	0·43	0·45	0·51	0·50	0·48

Besides these factors, the amount of soluble nitrogenous constituents will naturally vary with the temperature at which the malt is dried, etc., and with the manner in which the extraction is made. In the table of the results given by commercial malt extracts, Nos. 3 and 5 were stated by the manufacturers to have been overheated. They had a slightly burnt odour and were of a dark brown colour, though, curiously, they still possessed some diastatic power. The soluble nitrogen here amounted to only 0·40 per cent., whereas the usual amount is about 0·65 per cent.

The Carbohydrates.—As the initial action of the diastase takes place at a very low temperature, the transformation will almost invariably be that of the No. 8 equation, the yield being practically 80 per cent. of maltose and 20 per cent. of dextrin. Possibly some further degradation of the dextrin may take place if the wort is allowed to stand for any considerable length of time before concentration. No attempt has been made to determine the relative amount of these constituents; the amount of copper reduced has been simply calculated into maltose.

METHOD OF ANALYSIS.

Determination of the Total Solids.—Ten grammes of the sample were dissolved in water, made up to 100 c.c. and filtered. As a rule, the amount of insoluble matter was insignificant; when this was not the case it was estimated. The specific gravity of the solution was then taken at 15° C., and the amount of solid matter present found by reference to Schultze's table. The portion of this within which commercial malt extracts diluted in this way will be found to fall is given below. To save space, the intermediate values are omitted, but they may be found by interpolation:

SCHULTZE'S TABLE FOR DETERMINATION OF EXTRACT FROM THE SPECIFIC GRAVITY AT 15° C.

Specific Gravity.	Grammes in 100 C.c.	Specific Gravity.	Grammes in 100 C.c.
1·0250	6·60	1·0300	7·94
1·0255	6·75	1·0305	8·06
1·0260	6·88	1·0310	8·18
1·0265	7·03	1·0315	8·29
1·0270	7·18	1·0320	8·42
1·0275	7·32	1·0325	8·54
1·0280	7·46	1·0330	8·68
1·0285	7·58	1·0335	8·82
1·0290	7·70	1·0340	8·96
1·0295	7·82		

Determination of Phosphoric Acid.—This was made by the method first described by Hehner, the P_2O_5 being precipitated as phosphomolybdate, weighed as the ammonium compound, and the result divided by 28·5.

Determination of Albumose Nitrogen.—So far as we are aware, the statement of Bömer that $ZnSO_4$ can replace $(NH_4)_2SO_4$ has not been confirmed for malt extracts. We have tried "salting out" with both sulphates, and have obtained practically identical results:

	NITROGEN PRECIPITATED BY	
	$(NH_4)_2SO_4$.	$ZnSO_4$.
1.	0·09	0·085
2.	0·157 0·127	0·0148

Nitrogen precipitated by Bromine.—In some cases determinations were made of the N precipitated by Br in the filtrate from the $ZnSO_4$ precipitations by Allen's modification of Rideal and Stewart's method. It was found that the amount precipitated in this way closely approximated to that of the albumose nitrogen, and to that precipitated by phosphotungstic acid.

It should be mentioned, however, that a recent paper by Schjerning (ANALYST, xxvi., 77) shows that bromine precipitates certain non-proteid nitrogenous constituents, and that, on the other hand, it only precipitates part of the proteids of separated milk and blood serum.

Specific Rotatory Power.—This was determined in the 10 per cent. solution and calculated on the total solids. For clarifying, it was found that if a little kieselguhr was shaken up with the solution previous to filtration a brilliant filtrate was readily obtained.

Diastatic Power.—The malt extract made in the laboratory and employed in the estimation of the diastatic power of a malt is prepared by extracting 25 grammes of malt with 250 c.c. of water; it has a specific gravity of about 1013 (water = 1000), and contains about 3·4 per cent. solid matter. As the figures given for a commercial malt extract, this must necessarily be of a purely empirical nature. It was decided to dilute the sample under examination to a specific gravity of 1013—that is, to the same specific gravity as the malt extract used in determining the diastatic power of a malt, and to calculate the result in the same way as if examining a malt. This will give a rough estimate of the amount of diastatic power which has been lost during the manufacture of the extract where that of the malt is known and may at times prove useful. For example, a commercial malt extract was diluted until the resulting solution had a specific gravity of 1013; 1 c.c. of this was added to 100 c.c. of a 2 per cent. solution of soluble starch, and after the prescribed treatment the reduced copper weighed. A correction for the reducing bodies was then made by treating 1 c.c. of the diluted extract with 50 c.c. of Fehling's solution and 100 c.c. of water, the weight of reduced copper being deducted from that previously found, and the calculation made in the following manner:

$$\frac{0{\cdot}1314 - 0{\cdot}0219}{0{\cdot}438} \times 100 = 25 \text{ diastatic capacity.*}$$

Obviously, Lintner's tube method may also be employed, using for the determination of the reducing substances present four times the quantity of the diluted solution used in the first determination, and making allowance for this afterwards. The operation is, however, more tedious.

Composition of Commercial Malt Extracts.

The following table gives the complete results of the analyses of a series of malt extracts, also of several substances which might be used as adulterants:

	Water.	Total Solids.	Ash.	Phosphoric Acid (P_2O_5).	Total Nitrogen.	Albumose N.	N precipitated by Phosphotungstic Acid.	Ammoniated N.	Reducing Substances as Maltose.	Specific Rotation $[\alpha]_D$ of Solids.	Diastatic Capacity.
1. Wort evaporated on water-bath	18·80	81·20	1·60	0·67	0·69	0·09	—	0·05	—	97·2	—
2. Medicinal ...	19·90	80·10	1·04	0·49	0·70	0·15	0·103	0·09	59·5	109·8	21·6
3. French ...	19·9	80·1	1·62	0·50	0·42	0·098	0·21	0·021	51·04	117·3	12·0
4. " ...	18·2	81·8	1·62	0·52	0·79	—	—	0·032	52·3	110·2	10·0
5. "Scotch" ...	23·4	76·6	1·90	0·76	0·53	—	—	—	54·0	97·9	13·0
6. English medicinal ...	21·1	78·9	1·32	0·53	0·77	0·19	—	—	56·0	111·5	5·6
7. English medicinal	21·3	78·7	1·75	0·77	1·52 1·52	0·17	0·19	—	56·2	63·5	100·0
8. English medicinal ...	19·9	80·1	2·20	0·66	1·03	—	—	—	56·1	87·8	25·7
9. English medicinal ...	17·5	82·5	1·60	0·75	0·68	—	—	—	51·4	115·1	22·0
10. Baker's ...	24·4	75·6	2·10	0·79	0·98	0·14	—	—	52·56	85·9	24·0
11. "Diastase"	21·1	78·9	3·90	1·15	2·25	—	—	—	45·6	38·1	115·0
12. Glucose ...	15·8	84·2	0·55	0·065	0·20	—	—	—	62·8	128·2	—
13. Maltodextrin, "non-fermentable"	24·9	75·1	0·55	0·23	0·19	—	—	—	50·3	153·1	—
14. Dextrin syrup	18·0	82·0	1·0	0·24	0·30	—	—	—	55·68	134·1	—

Of the malt extracts given in this table, No. 1 was a sample of wort evaporated on the water-bath to a specific gravity of 1·330.

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* For fuller details of this method see the author's paper, ANALYST, xxi., 122.

Nos. 3 and 4 were of French origin. The manufacturers stated that these had been spoiled by overheating. They had rather a treacly odour, and were very dark in colour. The overheating would account for the low total nitrogen of No. 3, though, as already pointed out, their diastatic capacity had not been entirely destroyed. No. 5 was sold as "Scotch" malt extract, but we have reason to believe that it too was French.

Nos. 6 to 9 were all specimens of English medicinal extract. No. 7, which had the highest diastatic capacity of any of the extracts, excluding No. 11, is remarkable for its high total nitrogen.

No. 8, medicinal, No. 10, Baker's malt extract, and No. 11, "diastase," were all from the same manufacturer. They all showed a high ash and a low percentage of reducing substances. Probably some other process than simple evaporation had been used in their manufacture.

The "diastase" had a bitter taste and granular appearance. It contained a large amount of insoluble matter and a high percentage of nitrogen.

No. 12 was a specimen of ordinary glucose syrup; No. 13, "non-fermentable" dextrin syrup; and No. 14, a specimen of the so-called "malto-dextrin" syrup.

In conclusion, we wish to express our best thanks to the different manufacturers for the courteous manner in which they have placed samples of their preparations at our disposal, and also to Dr. L. T. Thorne and Mr. A. R. Ling for assistance in the experimental portion of the paper.

DISCUSSION.

Mr. HEHNER asked how the authors accounted for the very low specific rotatory power of sample No. 11, and also whether the result of their investigation was not to show that the diastatic power of these so-called malt extracts was very little, if any, greater than that of the malt itself.

Mr. BAKER said that malt extracts with comparatively high diastatic powers were obtained when the malt infusions were evaporated under reduced pressure. The diastatic power of the malt employed (on Lintner's scale) in such cases might be between 80 and 120, and that of the extracts between 120 and 500. Such extracts were used in many breweries where malt substitutes were employed; and they were also used as yeast foods. They were rich in the so-called maltopeptones—a recognised yeast recuperator.

Mr. CHAPMAN said that the following were the results of an analysis which he had made of a material of the class referred to:

Maltose	45·06
Dextrin	7·20
Invert sugar	14·49
Cane sugar	3·40
Ash	1·70
Water	21·76
Undetermined (including albuminoids, etc.)	6·39
	100·00
Specific rotatory power (of the sample itself)	+66·2

The large proportion of invert sugar was rather remarkable, though he did not know whether the sample was genuine or whether it contained added invert sugar. The ready formed sugars of the malt would account for some invert sugar, though probably not for so much as this sample contained.

Mr. Mitchell, in reply, said that the low specific rotatory power of sample No. 11 was probably due to the absence of carbohydrates and consequent increase in other substances. It would be noted that the proportion of total nitrogen in this sample was 2·25 per cent., representing a proportionately large quantity of proteids. Possibly, too, some method of precipitation—with ammonium sulphate, for instance—had been employed in addition to concentration, to increase the proportion of diastase in the sample. He could quite understand that, starting with a low-dried malt, extracts could be obtained of the high diastatic powers mentioned by Mr. Baker, though he had not himself met with any so high. It was not perfectly fair to compare the diastatic powers of these extracts according to Lintner's scale. The figures ought to be multiplied to correspond with the specific gravity of the extracts, which was much greater than that of a wort corresponding to the extract on which Lintner's determination was made.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

The Colouring Matter of Butter and Substitutes. J. Vandriken. (*Ann. Pharm.*, 1901, vii., 110; through *Chem. Zeit. Rep.*, 1901, 106.)—Stale amyl nitrite (*i.e.*, a product acid with nitrous acid) bleaches natural butter, but does not affect a factitious material or margarine unless it has been coloured with annatto. The test may be applied by treating 2 c.c. of filtered fat with an equal volume of ether, then adding 6 to 10 drops of the acid amyl nitrite and shaking; if the butter is pure, the colour should disappear at once. Or the ethereal solution may be agitated with 25 or 30 drops of "spirit of nitrous ether," when decolorization will be slower. Amyl nitrite bleaches annatto, hardly affects safranine tincture, does not change turmeric or carrot juice, and has no effect upon another (unknown) dyestuff largely used at present in the tinting of butter.
F. H. L.

(A number of coal-tar yellow colours are decolorized by acid nitrite, and such colours are used for colouring butter.
O. H.)

Examination of Pepper. A. L. Winton, A. W. Ogden and W. L. Mitchell. (*Twenty-second Annual Report of the Connecticut Experimental Station.*)—The authors have found that the amount of nitrogen in the fixed ether-extract is a useful datum in judging of the quality of pepper. They proceed as follows: Two grammes of the powdered sample are extracted with ether for twenty-four hours, the ether allowed to evaporate at the temperature of the room in a weighed dish, and the residue in the dish allowed to stand for eighteen hours over sulphuric acid. This gives the total ether-extract. The residue is then heated to 100° C., and kept at this temperature for six hours, when the temperature is raised to 110° until the weight is constant. This gives the fixed ether-extract. The amount of nitrogen is then determined in this extract by the Kjeldahl-Arnold process. The following table shows the percentage of nitrogen in the ether-extracts of various samples of pepper, etc.:

	Maximum.	Minimum.	Average.
Black pepper (14 samples ...	0·40	0·27	0·33
White pepper (11 samples) ...	0·34	0·26	0·30
Pepper hulls	—	—	0·09
Pepper hulls and dust	—	—	0·14
Long pepper	—	—	0·22
Buckwheat hulls	—	—	0·00

H. L.

The Valuation of Gum Arabic. O. Fromm. (*Zeit. anal. Chem.*, 1901, xl., 143-168.)—The author has made a series of investigations to determine the value of certain physical and chemical data as criteria of the value of a sample of gum as an adhesive agent.

Preparation of a Standard Solution.—In each case a solution of a specific gravity of 1·035 at 15° C., is prepared, this corresponds to about 10 per cent. of air-dried gum, and 8·5 grammes of anhydrous substance. An average sample of about 50 grammes is coarsely powdered, and shaken at intervals with 200 c.c. of water. The next morning it is filtered through muslin into a 500 c.c. cylinder, and diluted with water to a specific gravity of 1·035.

Frothing of the Solution.—The frothing properties, which all gums possess to a greater or less degree, is attributed by Wiesner (*Rohstoffe des Pflanzenreichs*, i., 85) to the presence of enzymes. In selecting a sample preference should be given to one which froths least, since frothing has an injurious effect on gumming machines.

Insoluble Matter.—The residue on the filter, consisting of mineral matter and substances which swell up on treatment with water without dissolving, is washed with water into a measuring cylinder, where the next day the amount of sediment is read. Its amount varies from an insignificant quantity to 50, 60, or 70 c.c. from 50 grammes of gum.

Viscosity of the Solution.—This is determined in Engler's viscosimeter at 20° C., and compared with the viscosity of water at the same temperature. As a rule, the results obtained by the author were about 2. The highest were 2·65-2·8 and 6·27, and the lowest 1·13.

The Acid Value.—Fifty c.c. of the standard solution are titrated with $\frac{N}{10}$ sodium hydroxide, and the results expressed in c.c. The figures thus obtained varied from

1·5 to 9·8 c.c., the mean being 2·1 c.c. The number of c.c. multiplied by 0·9112 gives the number of milligrammes of sodium hydroxide required to neutralize 1 gramme of the anhydrous gum. The factor 1·2731 gives the corresponding amount of potassium hydroxide.

Experiments made by the author show that the acidity of the solution steadily rises on keeping, and it is therefore advisable to invariably make the determinations at the same period, preferably on the day after the solutions have been made. The specific gravity of the solutions had not altered after keeping for a month, but the viscosity had decreased by an insignificant amount.

The Rotatory Power.—This is determined in a 100 millimetre tube. The author found the best varieties without exception to give a negative reading. The direct angle of rotation, a_D, rose as high as $-3°$ (corresponding to a specific rotation $[a]_D = -34°$). As a rule, it was between $-2°$ and $-3°$ (corresponding to $[a]_D$ $-23°$ and $-34°$). Rotations below $-2°$ and positive readings were exceptional.

Behaviour towards Reagents.—Nearly all the specimens examined by the author gave a thick precipitate with lead acetate, but in exceptional cases no precipitate was obtained with certain dark-coloured varieties, such as Amra'd or Gesirah gum.

The statement of the text-books that gums do not reduce Fehling's solution is stated to be absolutely false; the author almost invariably obtained a perceptible, though usually a slight, reduction.

Determination of the Tenacity.—Dalén's method is recommended for comparative determinations. The mean breaking strain and elasticity of a suitable absorbent paper are determined, and also the mean weight of strips, 18 centimetres × 15 millimetres, under the average conditions of atmospheric moisture (65 per cent.). Pieces of this paper are now placed on the solution of gum spread on a glass plate, and after removal of the excess of gum, dried in an atmosphere in which the moisture is 65 per cent. When dry, twenty strips are cut from the gummed paper, and the breaking strain again determined.

The method is illustrated by the following example :

The gummed paper had a mean breaking strain in one direction of	6·52 kilos	
And in another direction of	4·895 ,,	
Mean		5·7075
The ungummed paper had a mean breaking strain of...		2·902
Difference		2·8055
Ten gummed strips weighed	2·3555 grammes	
Ten ungummed strips weighed	2·0055 ,,	
	0·350 gramme.	

Therefore one strip contained 0·035 gramme of gum. The length of a hypothetical strip of gum 15 millimetres broad, weighing 2·8055 grammes, is then calculated from the proportion :

$$0{\cdot}035 : 0{\cdot}18 = 2{\cdot}8055 : x$$
$$\text{where } x = 14{\cdot}43 \text{ kilometres.}$$

The result thus obtained is used to express the degree of tenacity of the gum in a solution of specific gravity 1·035 and with a particular paper, and is only relative.

Elasticity.—It is well known that gummed paper has a tendency to curl up, and that when unrolled the layer of gum cracks. The author attributes this to the smaller degree of elasticity of the gum, for the greater the elasticity, as found in tearing experiments with gummed strips, the less the trouble experienced from this source. As a rule, the difference between the elasticity of the gummed and ungummed strips of paper expressed in percentages was somewhat over 2. Only inferior varieties gave values of less than 1·9, whilst in exceptional instances the values 1·51 to 1·68 were obtained.

Representative Analyses.—The following table may be given from the author's results as typical of good, medium, and bad varieties of gum :

Commercial Name.	Price per 100 Kilos.	Solution, Specific Gravity 1·035.	Frothing.	Insoluble Matter from 50 Grammes.	Viscosity at 20° C.	Acid Value $\frac{N}{10}$ NaOH.	Rotation a_D.	Action on Fehling's Solution.	Elasticity.	Tenacity.
	Marks			c.c.		c.c.			Per cent.	
Senegal...	170	very pale	moderate	35	2·41	2·3	−2° 47′	very slight reduction	2·13	14·8
Kordofan	190	pale	,,	15	2·17	4·45	−2° 29′	some reduction	2·10	15·2
,,	145	,,	slight	52	2·23	2·35	−2° 47′	very slight reduction	2·13	15·3
Senegal...	158	pale	strong	8	1·87	2·15	−2° 51′	perceptible reduction	2·19	13·0
,,	172·5	,,	,,	10	1·69	2·1	−1° 44′	some reduction	2·12	11·2
Kordofan	128	very pale	moderate	11	1·59	2·3	−2° 36′	perceptible reduction	2·10	11·3
Senegal...	143	yellowish	,,	35	1·9	2·0	−2° 27′	some reduction	2·15	12·7
*Senegal...	115·5	light brown	moderate	18	1·58	2·25	− about 1°	very slight reduction	1·99	11·0
,,	156	reddish-yellow	,,	6	1·4	1·7	+3° 21′	very slight reduction	1·85	10·5
*Amrad...	85	light brown	strong	3	1·66	1·9	−1° 12′	very slight reduction	1·96	10·9
*Gesirah...	90	reddish-brown	,,	7	1·3	1·9	+ about 3°	some reduction	2·09	9·8

From the examination of more than 300 samples, the author has found that, as a rule, gums with high tenacity also possess a high viscosity, high acid value, and high negative optical rotation. He gives the subjoined table of the results obtained with abnormal samples.

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* These gave no precipitate with lead acetate.

Commercial Name.	Price per 100 Kilos.	Solution, Specific Gravity 1·035.	Frothing.	Insoluble Matter from 50 Grammes.	Viscosity at 20° C.	Acid Value $\frac{N}{10}$ NaOH.	Rotation a_D.	Action on Fehling's Solution.	Elasticity.	Tenacity.
	Marks			c.c.		c.c.			Per cent.	
Arabicum	96	brown	very strong	—	2·34	9·8	+about 1°	no reduction	1·90	13·[illegible]
*Senegal...	121	,,	moderate	2	1·66	1·5	+9° 8′	very slight reduction	2·02	12·1
Gesirah...	120	reddish-brown	,,	8	1·28	2·05	+about 5°	some reduction	1·87	11·[illegible]
Senegal...	110	reddish-brown	,,	16	1·57	2·95	—	marked reduction	1·78	12·[illegible]
*Kordofan	110	reddish-brown	,,	5	1·13	1·6	—	perceptible reduction	1·51	9·3
,,	130	dark brown	very strong	15	1·21	1·3	—	some reduction	1·68	9·1
*Senegal...	121	pale	moderate	60	1·75	1·7	+8° 31′	some reduction	2·15	12·6
,,	88	pale, turbid	strong	65	6·27	2·1	−4° 52′	very slight reduction	2·22	12·9

C. A. M.

Volumetric Estimation of Morphine with Potassium Iodate and Alkaline Arsenious Acid. C. Reichard. (*Chem. Zeit.*, 1901, xxv., 328.)—This article consists principally of a discussion of the test for morphine in which potassium iodate and a mineral acid are used. It is known that a yellow or brown colour or a reddish-brown precipitate appears, and that on adding ammonia a very dark and quite permanent brown colour is produced. Based on this reaction, Reichard suggests a volumetric method of determining the alkaloid. The morphine, potassium iodate, and sulphuric acid are shaken together till the colour develops, the liquid and precipitate are extracted with chloroform (or less advantageously with carbon bisulphide), and the free iodine in the solution is titrated with alkaline arsenious acid. One atom of iodine is equivalent to 3 molecules of morphine hydrochloride, or 2 atoms of iodine to 3 of the sulphate.

Referring to his previous article (*Chem. Zeit.*, 1900, xxiv., 1061), Reichard now finds that an ammoniacal solution of silver chloride is to be preferred to silver nitrate, especially in dealing with morphine hydrochloride. The silver process is available for estimating the alkaloid in opium, further experiments thereon being in progress.

F. H. L.

Detection and Estimation of Morphine. F. Wirthle. (*Chem. Zeit.*, 1901, xxv., 291.)—Kippenberger has lately proposed a mixture of chloroform with 10 per cent. of alcohol as the immiscible solvent for recovering morphine from aqueous solutions, and he prescribed that it should be employed on a liquid which, originally alkaline, had been treated with a bicarbonate to destroy the alkali hydroxide. The process is not satisfactory; but if the solution is made slightly acid with hydrochloric acid and then alkaline with ammonia, Kippenberger's solvent works perfectly. For quantitative purposes, between five and nine extractions are necessary; and a small loss of water of hydration which occurs when morphine hydrochloride is dried for one hour, balancing a small quantity of foreign matter extracted by the chloroform, the results of the method are approximately correct. Marquis's quantitative process with ethyl acetate cannot be recommended.

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* These gave no precipitate with lead acetate.

Marquis's qualitative test for morphine—a violet colour* on adding a few drops of a reagent containing 3 c.c. of strong sulphuric acid and 2 drops of formalin—is not characteristic; but it is so delicate (showing fractions of a decimilligramme) and so easy to carry out, that it should be serviceable in examining aqueous liquids to see if they have been completely extracted as above. The various colours of the Pellagri test, which is so very characteristic of morphine, appear in the presence of several decimilligrammes of the alkaloid.

F. H. L.

Separation of the Active Constituents of Frangula, Sagrada, and Rhubarb. **E. Aweng.** (*Apoth. Zeit.*, 1901, xvi., 257; through *Chem. Zeit. Rep.*, 1901, 125.)—The material in fine powder is extracted with boiling water and an equal volume of 95 per cent. alcohol is added to the liquid. It is filtered and concentrated till its weight is one half that of the sample taken. When cold, enough alcohol is introduced to made the whole contain 80 per cent. of spirit by volume. The mixture is allowed to rest for several days, and the frangulic acid which separates is washed with absolute alcohol and ether, then dried over sulphuric acid. It forms a light brownish-yellow powder, easily soluble in 50 per cent. spirit, slightly soluble in water. The filtrate contains a double glucoside, a compound of frangulic acid with pseudo-frangulin. It is diluted with water and then evaporated, the product appearing as an amorphous red mass. This is soluble in water and in absolute alcohol; it is thrown down from the latter by dry ether in canary-yellow flakes. The compound splits up when its solution in 95 per cent. alcohol is treated with acetic acid, yielding frangulic acid and pseudo-frangulin, which dissolves in alkalis with a blood-red colour. The author finds in frangula bark 10·66 per cent. of the double glucoside and 3 per cent. of the acid; in sagrada, 14 and 3·7 per cent. respectively; in Shens-rhubarb, 35·5 and 1·8 per cent.; and in the *R. rhaponticum*, 29·4 and 3·8 per cent.

F. H. L.

ORGANIC ANALYSIS.

The Quantitative Estimation of Hydrogen Sulphide in Illuminating Gas. **C. C. Tutwiler.** (*Journ. Am. Chem. Soc.*, xxiii., 173.)—The following method gives good results when a rapid estimation of hydrogen sulphide is required, as in testing the efficiency of a purifier. The gas is passed through a burette of special construction until the latter is filled with it, and mercury then run in from a levelling bulb until 100 c.c. of gas at atmospheric pressure remain in it. The mercury is then withdrawn so that the gas is under diminished pressure and exactly fills the burette, which is disconnected from the mercury reservoir. Five c.c. of starch paste are then sucked in, and then drop by drop, with constant shaking, a standard solution of iodine, which is supplied from a graduated measure fitted to the upper part of the gas burette, until the blue colour is permanent. Three grains of hydrogen sulphide in 100 cubic feet of gas can be detected in this way. A. G. L.

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* Cf. Kobert, ANALYST, 1899, xxiv., 241.

Dermination of Sulphur in Benzol for use in Gas-works. W. Irwin. (*Journ. Soc. Chem. Ind.*, 1901, 440.)—The method is an adaptation of Allen's test for sulphur in fats, and is carried out in the ordinary apparatus for estimating sulphur in gas, the burner being replaced by a small lamp. Of the benzol to be tested 10 c.c. are measured into the spirit-lamp, 90 c.c. of alcohol are added, and the determination is carried on as usual. A. M.

Estimation of Indican in Urine by means of Isatin and Hydrochloric Acid. J. Bouma. (*Zeits. physiol. Chem.*, 1901, xxxii., 82; through *Chem. Zeit. Rep.*, 1901, 131.)—The urine is precipitated with one-tenth its volume of lead acetate, the filtrate is mixed with an equal bulk of a solution of isatin in hydrochloric acid, and the whole is heated on the water-bath for fifteen minutes till it becomes (usually) dark red. When cold, it is extracted with chloroform, the solvent removed, and the residue dried for two hours at 110° C. The mass is next completely extracted with hot water to dissolve the excess of isatin (which has passed into the chloroform and would absorb permanganate in the final titration), and dried again. It is then treated with sulphuric acid and titrated with permanganate, calculating the result as indigo-red disulphonic acid. For clinical purposes a colorimetric process gives approximate accuracy: 20 c.c. of urine are treated with 2 c.c. of lead acetate and filtered through a dry paper; 5·5 c.c of the filtrate are mixed in a tube with 5 c.c. of a solution of 20 milligrammes of isatin in 1 litre of HCl (this 5 c.c. is sufficient for urines containing indoxyl equivalent to 20 milligrammes of indigo). The liquid is boiled for a few seconds, cooled, well shaken with 5 c.c. of chloroform, and the colour which develops is compared with a set of standards prepared with indigo-red.

From numerous experiments the author finds that the yield of indigo-blue is considerably less than one half that of indigo-red, but that there is no fixed relation between them. It appears also that the yield of indigo-red as above is rather more than double that of the total indigo pigments when the ferric chloride and hydrochloric acid method is adopted. This peculiarity is explained by a more perfect conversion of the indoxyl into indigo when using the isatin process. F. H. L.

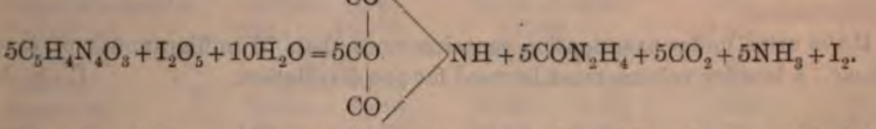
The Determination of Uric Acid in Urine. H. Bouillet. (*Bull. Soc. Chim.*, 1901, xxv., 251-255.)—This method is based upon the fact that iodic acid decomposes uric acid, yielding in the first place alloxan and urea:

$$C_5H_4N_4O_3 + O + H_2O = C_4O_4N_2H_2 + CON_2H_4.$$

The alloxan is then hydrolysed, and there is a liberation of carbon dioxide and of ammonia, which combines with the excess of iodic acid:

$$C_4O_4N_2H_2 + H_2O = CO_2 + NH_3 + C_3O_3NH.$$

The final equation, which the author states has been confirmed by all his determinations, is:



$$5C_5H_4N_4O_3 + I_2O_5 + 10H_2O = 5\begin{matrix}CO\\ | \\ CO\\ | \\ CO\end{matrix}\!\!>\!NH + 5CON_2H_4 + 5CO_2 + 5NH_3 + I_2.$$

The uric acid is precipitated as barium urate by adding barium chloride to 100 c.c. of the urine, previously neutralized with sodium hydroxide, until no more precipitate is formed. The liquid is then acidified with 5 c.c. of acetic acid (1 : 100), and left for fifteen to twenty minutes, after which the precipitate, consisting of urate, phosphate and sulphate of barium, is collected and washed.

It is next washed with a jet into a porcelain dish, the total volume of liquid and precipitate being 100 to 150 c.c. The uric acid is liberated by the addition of 20 c.c. of sulphuric acid (1 : 10), and the liquid heated to the boiling-point. At that moment 10 c.c. of a standard solution of iodic acid are introduced, and the boiling continued until all the iodine has been expelled. When cold the residual iodic acid is determined by adding 10 c.c. of hydrochloric acid (1 : 10), 30 c.c. of potassium iodide solution (1 : 10), and titrating the liberated iodine with $\frac{N}{10}$ thiosulphate. The difference between the amount of iodic acid taken and left, multiplied by 0·007, gives the amount of uric acid. The accuracy of the results thus obtained is illustrated by test experiments. C. A. M.

A Gas-Volumetric Method of Determining Acetone in Urine. **E. Riegler.** (*Zeit. anal. Chem.*, 1901, xl., 94-97.)—This method is based on the facts that if phenyl-hydrazine be shaken with hot Fehling's solution, the whole of its nitrogen is liberated, but that if acetone be present a hydrazone is produced which is not decomposed by Fehling's solution.

$$CH_3.CO.CH_3 + H_2N - NH.C_6H_5 = \begin{matrix}CH_3\\ CH_3\end{matrix}\!\!>\!C = N - NH.C_6H_5 + H_2O.$$

The reagents required are: (1) A solution of 1 gramme of crystallized phenyl-hydrazine hydrochloride in 50 c.c. of water, filtered if necessary; (2) a 15 per cent. solution of copper sulphate; and (3) a 15 per cent. solution of sodium hydroxide.

In the author's method 10 c.c. of the phenyl-hydrazine sulphate solution are placed in the exterior evolution flask of a Knop-Wagner nitrometer, 40 c.c. of water added, and 10 c.c. of the sodium hydroxide solution introduced. Ten c.c. of the copper sulphate solution are placed in the inner vessel of the apparatus, and after ten minutes the flask is thoroughly shaken, and the liberated nitrogen collected and measured.

In determining the acetone in urine, 50 c.c. of the latter are mixed with 1 c.c. of glacial acetic acid and distilled, the distillate being collected in a flask containing

10 c.c. of the phenyl-hydrazine solution and about 1 gramme of crystallized sodium acetate. The distillation is continued until 40 to 45 c.c. of the urine have passed over.

The flask is heated for about fifteen minutes on the water-bath. On cooling, its contents are introduced into the nitrometer, and the nitrogen liberated as in the blank experiment. The difference between the amounts of nitrogen at standard temperature and pressure, multiplied by the factor 2·6, gives the quantity of acetone in milligrammes.

If the urine under examination contains more than 70 milligrammes of acetone in 50 c.c., a smaller volume must be used for the distillation. C. A. M.

The Determination of Free Hydrochloric Acid in the Gastric Juice. Meunier. (*Journ. Pharm. Chim.*, 1901, xiii., 367-369.)—The method proposed by the author is a rapid modification combining those of Gunzbourg and Robin.

Five c.c. of the gastric juice are titrated approximately with $\frac{N}{10}$ sodium hydroxide solution with one drop of Toppfer's reagent (dimethyl-amido-nitro-benzene—D A A B) as indicator. It is difficult to determine the exact point of the change in colour from rose to reddish orange. Hence, from 0·1 to 0·5 c.c. too much alkali is usually added. Let 3 c.c. represent the amount used in a typical determination.

A fresh portion of 5 c.c. of the gastric juice is next titrated with $\frac{N}{10}$ sodium hydroxide, commencing with 2·6 c.c. and adding successively 0·1 c.c. until the 3 c.c. used in the first titration is reached. After each of these additions one drop of the liquid is heated in a porcelain crucible on the water-bath at 60° C., with one drop of Gunzbourg's reagent (phloroglucinol, 2; vanillin, 1; and 80 per cent. alcohol, 100 parts).

After a few minutes the five crucibles are examined. If the contents of the first alone are red, 2·6 c.c. of $\frac{N}{10}$ sodium hydroxide solution are required to neutralize the free hydrochloric acid. If the first, second, third, and fourth, are all red, 2·9 c.c. are required, and so on. C. A. M.

A Comparison between the Bromine and Iodine Absorption Figures of Various Oils. H. T. Vulte and Lily Logan. (*Journ. Amer. Chem. Soc.*, xxiii., 156.) —The authors have carefully revised the bromine and iodine figures for various oils. In the case of bromine, the loss due to the formation of substitution products was also determined by means of the potassium iodate method. The iodine value was determined by the Hübl method; the bromine figure almost exactly as proposed by McIlhiney (*Journ. Amer. Chem. Soc.*, xxi., 1084), except that twenty to thirty minutes were allowed to elapse before the titration. The substitution figure was determined as soon as possible, since the addition products decompose readily. In the following table the oils are divided into five classes, the first one consisting of oils for which the two values are nearly the same. This is probably the case with all vegetable oils of simple constitution:

	Iodine.	Iodine calculated from Bromine.	Difference of Averages.	Difference of nearest Figures.
I. Olive oil	79·70— 80·40	80·31	0·26	0·09
Cotton-seed oil ...	97·50	97·41—97	0·295	0·09
Poppy-seed oil ...	127·98—128·55	128·37	0·055	0·28
Linseed oil ...	155·12—155·52	154·80	0·52	0·32
Sweet almond oil	90·53— 89·64	90·20— 89·64	1·08	0·33
Peanut oil ...	100·71—100·76	101·26	0·523	0·50
Whale oil ...	128·00	127·45—127·38	0·585	0·50
Lard oil	76·99— 77·36	76·03— 75·88	1·22	0·96
II. Sperm oil ...	79·95— 79·76	82·39— 82·08	2·422	2·13
III. Rape oil	103·69—103·37	99·03— 98·56	4·60	4·17
Castor oil ...	86·32— 87·15	78·74— 78·71	8·01	7·58
IV. Seal oil	93·31— 93·99	103·09—103·74	9·765	9·10
Cod-liver oil ...	122·09—122·79	132·86—132·18	10·08	9·39
Menhaden oil ...	176·5 —175·65	186·94—186·86	10·825	10·36
V. Resin oil ...	59·67— 58·95	10·60— 10·26	48·845	48·35

The following table gives the substitution figures for all oils examined which formed substitution products. Some of the oils were rancid, and it is probably due to this fact that sweet almond oil showed substitution :

	Total Bromine.	Addition Figure.	Substitution Figure.	Average Substitution Figure.
Cod-liver oil ...	85·16— 84·67	84·52— 84·09	0·63—. 0·58	0·605
Menhaden oil ...	120·10 – 120·30	118·88—118·93	1·22— 1·27	1·245
Sweet almond oil	59·54— 59·15	57·38— 57·02	2·16— 2·13	2·145
Sperm oil ...	54·61— 54·56	52·41— 52·21	2·20— 2·34	2·27
Castor oil ...	52·62— 52·80	50·09— 50·07	2·53— 2·73	2·63
Resin oil ...	108·67—109·23	6·74— 6·47	101·93—102·76	102·345

A. G. L.

On the Heat of Combustion as a Factor in the Analytical Examination of Oils, and the Heats of Combustion of some Commercial Oils. H. C. Sherman and J. F. Snell. (*Journ. Amer. Chem. Soc.*, xxiii., 164.)—Although the heats of combustion of fatty oils cannot be used as a means of distinguishing between them, since the variations are too small, the determination is of value in comparing oils rich in hydroxyacids with waxes or non-fatty oils. The oxidation consequent on drying also leads to a loss of calorific power, and this is sometimes of more value than the determination of increase of weight, which may be vitiated by the presence of some volatile oil like petroleum. In some drying experiments with linseed oil, a loss of 10 per cent. of calorific power was found, whilst lard subjected to the same treatment lost less than 1 per cent.

The heats of combustion given below were obtained with a bomb-calorimeter, and are the means of at least two determinations, the average deviation from the mean being $\pm 0{\cdot}12$ per cent. To reduce the values found at constant volume to heats of combustion at constant pressure,

$$\tfrac{1}{2}\,(p-g)\,\frac{T}{M}\ \text{calories per gramme}$$

were added, p being the number of atoms of hydrogen, and g the number of atoms of oxygen present in the molecule, M the molecular weight, and T the absolute temperature of the calorimeter. The values for the specific gravity, iodine absorption, and acidity were determined as usual :

Description of Oil.	Specific Gravity, $\frac{15\cdot5^\circ}{15\cdot5^\circ}$	Iodine Absorption. Per Cent.	Free Acid as Oleic. Per Cent.	Heat of Combustion, per Gramme.	
				Constant Volume. Calories.	Constant Pressure. Calories.
Raw linseed, I., 1900, fresh	0·934	182·4	4·30	9,364	9,379
,, II., 1898	0·938	175·9	1·22	9,379	9,394
,, III., old	0·947	156·7	5·30	9,215	9,230
Boiled linseed	0·953	150·7	7·40	8,810	8,824
Poppy-seed	0·926	129·6	2·66	9,382	9,397
Maize oil, I., 1900	0·924	120·3	3·32	9,413	9,428
,, II., 1898	0·926	120·7	2·56	9,436	9,451
,, III., crude	0·926	122·4	1·68	9,419	9,434
Cotton-seed, I., prime yellow	0·920	102·5	0·20	9,396	9,411
,, II., choice yellow	0·921	106·4	0·32	9,401	9,416
,, III., choice white	0·923	105·5	0·08	9,390	9,405
,, IV., crude	0·927	103·2	2·28	9,397	9,412
,, V., crude	0·927	—	—	9,336	9,351
,, VI., rather old	0·929	100·1	0·92	9,323	9,338
,, VII., old	0·941	93·7	2·03	9,168	9,183
Sesame	0·924	105·3	1·65	9,395	9,410
Rape-seed, I.	0·922	107·4	0·82	9,489	9,504
,, II.	0·920	108·6	0·68	9,462	9,477
,, III.	0·926	99·3	2·94	9,412	9,427
Castor, I.	0·967	84·1	0·26	8,863	8,877
,, II.	0·964	86·9	2·18	8,835	8,849
Pea-nut (arachis)	0·917	105·9	0·16	9,412	9,427
Almond, I.	0·919	98·1	5·13	9,454	9,469
,, II.	0·931	89·8	7·00	9,311	9,326
Olive, I.	0·917	85·1	2·51	9,457	9,472
,, II.	0·916	78·8	0·40	9,451	9,466
Menhaden, refined	0·935	—	0·36	9,360	9,375
,, crude	0·934	—	1·92	9,371	9,386
Cod-liver, fresh	0·927	165·6	0·56	9,437	9,452
,, old	0·938	137·3	1·50	9,277	9,292
Whale	0·924	126·6	0·60	9,473	9,488
Lard oil, I., 1900	0·917	74·3	0·74	9,451	9,466
,, II., 1899	0·919	72·5	1·25	9,447	9,462
,, III.	0·922	72·9	2·64	9,394	9,409
,, IV.	0·924	69·3	2·34	9,372	9,387
Sperm oil	0·886	78·7	0·78	9,946	9,964
Rosin oil	0·989	76·9	14·40	10,145	10,159
Lubricating petroleum, I.	0·881	—	—	10,797	10,819
,, ,, II.	0·897	—	—	10,753	10,775
,, ,, III.	0·905	—	—	10,682	10,704

The variations of the heats of combustion appear to be more closely related t those in the specific gravities than to those in the iodine or acidity values. Th product of large calories into specific gravity at 15·5° is nearly constant at 8·80 t 8·63 for the fatty oils. Castor and boiled linseed oils show lower; sperm mineral, and rosin oils higher values. By dividing the heat of combustion by the specific gravity, a nearly constant value of 10·0 to 10·3 is obtained for the fatty oils, whilst sperm and mineral oils show a much higher value (11·2 for sperm oil and 11·8 to 12·3 for mineral oils). A. G. L.

Calorimetric Determination of Heating Value. K. Kroeker. (*Zeits. angew. Chem.*, 1901, 111.)—A criticism of Langbein's paper (ANALYST, 1901, 81). The correction introduced by Langbein for sulphur does not eliminate error, but introduces it, for in the bomb it is not all burnt to trioxide, but some sulphur dioxide is formed; on the other hand, in the furnace some trioxide is formed. The author also gives a description of the calorimetric bomb now used by him. This is made either with enamelled inner surface, or is coppered, and platinum foil is then soldered on. A. M.

Ammonium Persulphate as a Reagent for Alkaloids. N. A. Orlow and P. K. Horst. (*Farmaz. J.*, 1901, xl., 91; through *Chem. Zeit. Rep.*, 1901, 105.)—Ammonium persulphate gives the following reactions with alkaloids: Cocaine, a white precipitate soluble in excess, separating as a thick colourless liquid at the bottom of the tube; strychnine, a white pulverulent precipitate. In presence of sulphuric acid, chelidonine gives a yellow colour changing to green and then brown; chelerythrine, a violet to black; sanguinarine, dark brown. Corydaline, yellow, dirty green, dirty yellow. Bulbocapnine, blue then violet. Morphine, pale orange. Codeine, orange. Narcotine, orange red. Papaverine, yellow. Narceine, violet, blood-red, yellow. Apomorphine, green to blue. No colours are produced by cocaine, pilocarpine, caffeine, aconitine, colchicine, colchiceine, and quinine. F. H. L.

Chrome-Hide Powder for Analysis of Tanning Materials. B. Weiss. (*Gerber*, 1901, xxvii., 29; through *Chem. Zeit. Rep.*, 1901, 105.)—This modification of ordinary hide powder is prepared as follows: 100 grammes of powder are shaken up with 2 litres of water, and treated with (altogether) 3 grammes of chrome alum dissolved in 100 c.c. of water on three successive days. The product is filtered, and then washed by alternate agitation with fresh water and filtration till sulphuric acid can no longer be detected, 10 c.c. of 40 per cent. formalin being added to the last quantities of water. Finally, the hide powder is lightly pressed, rubbed down in the hands, and preserved in the damp. The material keeps well, and it is only necessary to determine the moisture in it occasionally. In carrying out an analysis of a tanning material, 130 c.c. of its solution are well agitated with that weight of treated hide powder which corresponds to 7 grammes of the dry product; the whole is allowed to rest overnight, then filtered, and the insoluble matter dried and weighed as usual. F. H. L.

Determination of Cyanic Acid in Commercial Cyanides. O. Herting. (*Zeits. angew. Chemie*, 1901, 585.)—The cyanide is dissolved in water, dilute hydrochloric acid or sulphuric acid is added, and the liquid is evaporated to dryness in a water-bath. The cyanate is decomposed according to the equation:

$$KOCN + 2HCl + H_2O = KCl + NH_4Cl + CO_2.$$

The ammonia produced is determined by distilling with excess of soda in the usual way, and the percentage of cyanic acid is calculated from it. The cyanide should of course be tested for ammonia, and if it be present it should be determined and allowed for in calculating the cyanic acid. A. M.

The Classification of Acidimetric and Alkalimetric Indicators. Julius Wagner. (*Zeit. Anorg. Chem.*, xxvii., 138.)—About two years ago F. Glaser divided indicators into three groups (*Zeit. Anal. Chem.*, xxxviii., 273). His division was based not on the chemical character of the indicators, but on their delicacy. His first group embraces feeble basic and actually acid substances, delicate towards alkalies, but not towards acids, *e.g.*, tropœolin OO, methyl-orange, congo-red. The second group includes acid indicators, mostly phenols, delicate towards feebler acids, but less sensitive towards bases, *e.g.*, fluoresceïn, litmus, *p*-nitrophenol. The third group consists of indicators even less acid, and consequently sensitive towards very feeble acids, but only reacting with the strongest alkalies, *e.g.*, turmeric, phenolphthaleïn, Poirrier's blue.

The author of the present paper prefers to class indicators according to their chemical character, having regard, in the first instance, to the manner in which they form ions, and then to form subgroups according to their delicacy, as follows:

A. Indicators with a monovalent characteristic ion: (1) with an anion; (2) with a kation.

B. Indicators with a polyvalent ion: (1) with positive and negative ions; (2) with mono- and di- valent anion or kation.

The two main groups are distinguished by the appearance or non-appearance of an intermediate colour, which, under certain conditions, allows of the determination of different anions or kations by means of the same indicator.

Indicators belonging to group A (1) are only slightly dissociated in acid solution. Their dissociation increases with the disappearance of the hydrogen ions, and is slower the feebler the acid nature of the indicator. Consequently, in this case strong bases must be used for the titration, whilst for weaker bases a more acid indicator must be used. The majority of indicators belong to this group, *e.g.*, fluoresceïn, *p*-nitrophenol, turmeric, litmus, phenolphthaleïn, and Poirrier's blue.

Group A (2) only contains two indicators, viz., methyl violet and amido-azo-benzol.

Indicators belonging to group B (1) form differently coloured ions in acid and alkaline solution, and their behaviour may be likened to that of aluminium hydroxide. An intermediate colour is consequently possible, and has been actually observed in the case of methyl-orange, and of a few others. All are strongly acid, and can be used even for weak bases like ammonia. Tropœolin OO, methyl- and ethyl-orange, and congo red are members of this group.

All the indicators included in group B (2) are acid, and resemble phosphoric acid in their behaviour. They permit of the titration of two acids present together, provided that one is much weaker than the other. Alizarin-sulphonic acid, which is yellow in acid solution, and turns first red and then violet on treatment with alkali, is an example of this group. Galleïn and phenacetolin also belong to it.

In group A (1) a new indicator seems to be required, which would give a sharp end reaction when a carbonate is titrated with a strong acid. In group B (2) also an indicator is wanted which would give good results when two acids are present together. A. G. L.

Occurrence of Zinc in Plants. L. Laband. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1901, 489.)—The author has estimated the amount of zinc in plants grown on soils containing a considerable quantity of zinc salts. The plants, which appeared quite healthy, were, after cleaning, dried at 100° C.; 100 grammes of the dry substance were heated with concentrated sulphuric acid and mercury until the solution was colourless. On dilution with water a little calcium sulphate crystallized out. Sulphuretted hydrogen was then passed through the strongly acid solution, the mercury sulphide filtered off and the excess of sulphuretted hydrogen driven off on the water-bath. After separating the phosphoric acid in the filtrate by means of tin and hydrochloric acid, the zinc was precipitated with sulphuretted hydrogen in acetic acid solution. The precipitate of zinc sulphide was filtered, washed with acetic acid, dissolved in hydrochloric acid, again thrown down as sulphide, and finally weighed as oxide. A hundred grammes of dry plants gave 0·252 gramme of zinc oxide. A second estimation, in which the zinc was determined electrolytically, gave 0·202 per cent. of zinc. W. P. S.

A Modification of the Method of Titrating with Permanganate. J. Gailhat. (*Bull. Soc. Chim.*, 1901, xxv., 395-401.)—The author has found that in the presence of an excess of manganous sulphate ($MnSO_4$) and of 20 per cent. by volume of sulphuric acid, solutions of potassium permanganate only lose on boiling a very slight trace of the oxygen which acts upon oxalic acid.

The amount of this loss depends upon the acidity of the liquid and of the concentration of the permanganate solution. If, however, the strength of the solution does not exceed 15 grammes per litre, the loss remains constant, whether the duration of boiling be five minutes or an hour.

It is thus possible to titrate substances which are readily oxidized under these conditions.

A definite volume of permanganate solution is mixed with an excess of manganous sulphate, and rendered acid with sulphuric acid. It is boiled for five minutes, and then titrated with a standard solution of oxalic acid.

An equal volume of the same permanganate solution, etc., is also boiled for five minutes, after which a weighed quantity of substance under examination is introduced, the boiling continued for an hour under a reflux condenser, and the liquid titrated as before. The difference between the two titrations gives the oxygen consumed by the substance.

The author has obtained very concordant results by this method in the analyses of crude and commercial glycerins. C. A. M.

INORGANIC ANALYSIS.

Determination of Arsenic in Coke. L. Archbutt and P. G. Jackson. (*Journ. Soc. Chem. Ind.*, 1901, 448.)—The coke is dried at 100° C., 50 grammes are taken and heated for at least two hours with 100 c.c. of pure nitric acid (1·42). Water is added to 400 c.c., and after settling the coke is filtered off on a toughened filter and washed. The filtrate is evaporated to dryness on a water-bath, the residue is mixed with 25 c.c. of concentrated sulphuric acid and heated with stirring. The heating is continued until fumes of sulphuric acid come off. Cold water is added to dissolve, and then it is again evaporated until fumes come off once more. This is repeated at least three times, till all the nitric acid is driven off. The residue is then transferred to a distillation-flask fitted with a thermometer and a condenser, the tube of which dips below the surface of about 20 c.c. of water contained in a receiving flask. Twenty-three grammes of sodium chloride and 2 grammes of ferrous sulphate are added to the liquid in the flask, which should be quite cold, and not more than 100 c.c. in volume. Distillation commences at about 110° C., and is continued until the temperature reaches 125° C., when the whole of the arsenic will be found in the distillate. To the latter is added a little zinc sulphide, and it is agitated for a few minutes until the precipitate has coagulated. The precipitate is filtered on a hardened filter, washed with sulphuretted hydrogen water, rinsed into a flask, boiled with 300 c.c. of water, the filter-paper is added, and boiling is continued another hour. The liquid is filtered, concentrated to about 40 c.c., cooled, ½ gramme of sodium bicarbonate is added and some starch solution, and the liquid is rapidly titrated with centinormal iodine solution. Practice should be acquired by titrating sulphide of arsenic precipitated from known quantities of a standard solution. A blank determination should be made. A. M.

Arsenic in Coal and Coke. R. F. Wood Smith and R. L. Jenks. (*Journ. Soc. Chem. Ind.*, 1901, 437.)—The fuel is burnt in a combustion-tube about 20 inches long. About 4 inches from one end this is constricted to a diameter of about 2 millimetres. The next inch is left to form a bulb, and the last 3 inches are drawn out and led into a wash-bottle containing sulphuric acid. Five to 15 grammes of the fuel are weighed out and introduced into the tube and burnt in a current of air, the products of combustion being passed through the wash-bottle. The heating lasts about three hours. When the tube is cold the ashes are withdrawn, and laid aside for the determination of "fixed" arsenic, whilst the contents of the bulb are washed into the bottle by means of a pipette long enough to reach down to the constriction, or the tube may be severed at the constriction. The ashes are triturated in a mortar, washed with dilute sulphuric acid into a flask, warmed for a few minutes, and then allowed to stand at least twelve hours. The liquid is then diluted, an aliquot part is taken, heated in a water-bath and passed into the Marsh apparatus. The contents of the wash-bottle are also made to a definite bulk, and an aliquot part is transferred to the Marsh apparatus. The latter portion is returned as "volatile arsenic." A. M.

The Titration of Arsenious Acid with Potassium Permanganate. O. Kühling. (*Berichte*, 1901, xxxiv., 404-406.)—If arsenious acid be dissolved in a solution of potassium hydroxide, and a sufficient quantity of zinc sulphate added, permanganate solution is decolorized, even in the cold. But when from 70 to 80 per cent. of the arsenious acid has been oxidized, the reaction proceeds very slowly, and it is necessary to add the permanganate in small amounts (not more than 0·2 c.c.), and to heat the beaker for fifteen to thirty minutes on the water-bath after each addition, until finally the red colour persists after thirty minutes' heating.

The potassium hydroxide should be purified by crystallization from alcohol, and the aqueous solution freed from alcohol by repeated evaporation on the water-bath.

If an excess of alkali be avoided, an initial addition of 20 c.c. of 10 per cent. zinc sulphate solution is sufficient, a little more being added when the oxidation process becomes too slow. In this reaction $2KMnO_4$ (=3O) oxidize $\frac{3}{2}$ As_2O_3.

As this method is too tedious for ordinary use, the author has devised the following method of accelerating the oxidation: The arsenious acid is dissolved in hot sulphuric acid (30 to 40 per cent. strength), in a flask previously cleansed with permanganate and sulphuric acid. The solution is diluted to 100 c.c., and heated to the boiling-point, whilst potassium permanganate solution is introduced, rapidly at first, and subsequently more slowly. After each addition the liquid is heated for one to two minutes nearly to the boiling-point, this process being continued until the red colour no longer disappears.

In this method $2KMnO_4$(=5O) correspond to $\frac{5}{2}$ As_2O_3.

The following results are typical of those obtained by these methods:

	Taken As_2O_3 gramme.	Found, gramme.
I.	0·2158	0·2171
	0·2171	0·2177
II.	0·2173	0·2177
	0·2178	0·2183

C. A. M.

Valuation of Gold Sodium Chloride. Johnson and Sons. (*Journ. Soc. Chem. Ind.*, 1901, xx., 210.)—Fifteen grains of the double chloride are dissolved in 1 ounce of water, the gold is precipitated with sulphurous acid, filtered off, ignited and weighed. Simultaneously 7 grains of pure gold are dissolved in aqua regia, evaporated, redissolved in water, precipitated, etc., exactly as before, using filters of the same weight. The object of this blank test is to fix the amount of loss in the process, and a corresponding addition is made to the yield of metal from the gold sodium chloride. The results are correct to the second place of decimals. F. H. L.

Notes on the Precipitation of Metals with Sodium Thiosulphate. E. Donath. (*Zeit. anal. Chem.*, 1901, xl., 141-143.)—It was shown by Wolcott Gibbs (*Zeit. anal. Chem.*, iii., 386) that sodium thiosulphate precipitated many metals as sulphides from their boiling solutions in hydrochloric or sulphuric acids. The author has studied the behaviour of the reagents with acetic acid solutions, and has observed the following facts:

When a solution of cadmium chloride or sulphate is treated with ammonia in excess, and then rendered strongly acid with acetic acid, the cadmium is precipitated quantitatively by adding powdered thiosulphate to the boiling liquid and continuing the boiling for thirty minutes, with the occasional introduction of a few drops of acetic acid.

When a solution of zinc is treated in the same way, there is a precipitate of sulphur, which only contains traces of zinc. A quantitative separation, however, is not possible, since in the presence of cadmium considerably larger quantities of zinc sulphide are precipitated.

Solutions of nickel and cobalt salts behave in an analogous manner, nickel being quantitatively precipitated, whilst pure cobalt solutions give no precipitate except finely-divided sulphur. If, however, a nickel solution be mixed with a solution of cobalt or of manganese, or iron sulphate, the nickel sulphide will contain more or less of the sulphides of these metals.

It is thus possible to detect minute traces of nickel by the fact that when present in a solution of cobalt a precipitate of nickel sulphide contaminated with cobalt is obtained. C. A. M.

Determination of Titanic Acid in Titanium Iron Ores. J. H. L. Vogt. (*Stahl und Eisen.*, xxi., 283; through *Zeitschrift für angew. Chem.*, 1901, 550.)—According to the author, 2·5 grammes of the ore are dissolved in hydrochloric acid, and potassium sodium carbonate is added to the insoluble residue. Silicic acid is separated in the usual manner, and is weighed and treated with hydrofluoric and sulphuric acids. The remaining titanic acid is brought into solution by fusing with acid potassium sulphate. By the addition of ammonia and bromine oxide of iron, alumina, manganese peroxide, phosphoric acid and titanic acid are precipitated. They are filtered and weighed. They are then dissolved in hot concentrated hydrochloric acid, the solution is exactly neutralized, then acidified with a few drops of sulphuric or hydrochloric acid. The ferric salts are reduced with sulphurous acid and the solution is boiled for at least one hour, thus precipitating titanic acid mixed with some alumina, phosphoric acid and a little oxide of iron. The liquid is filtered through a double filter, and the filtrate is again treated in the same manner. The residue is melted with pure soda for a considerable time over the blow-pipe to convert the alumina and phosphoric acid into soluble aluminates and phosphates, whilst the titanic acid forms sodium titanate insoluble in cold water. The latter is filtered off, washed with cold water, dissolved in hydrochloric acid, and after neutralization and the addition of sulphurous acid, the titanic acid is precipitated by boiling as described above. A. M.

Determination of Calcium in High-grade Ferro-silicon. G. W. Gray. (*Journ. Soc. Chem. Ind.*, 1901, 538.)—One gramme of the finely-ground sample is fused with about 4 grammes of potassium carbonate. The mass is dissolved out of the crucible as far as possible with water and hydrochloric acid, and the remainder is removed by fusing with potassium bisulphate. The iron is brought into solution with nitric acid. Silica, iron, calcium, etc., are separated and determined in the usual way.

A. M.

Rapid Estimation of Prussian Blue in Spent Oxide. J. M. Popplewell. (*Journ. Soc. Chem. Ind.*, 1901, xx., 225.)—Five grammes of the sample are boiled for five minutes with 50 c.c. of 5 per cent. sodium hydroxide. The filtrate is brought to the boil, mixed with 50 c.c. of 5 per cent. ferric chloride solution, and acidified with hydrochloric acid. If the precipitate of Prussian blue and sulphur appears in a finely-divided state, giving the solution a pale blue colour, more ferric chloride is added, till either the liquid is dark red (thiocyanates) or till the precipitate settles. This is filtered off and agitated for ten minutes in the cold with 50 c.c. of 5 per cent. caustic soda; filtered again, and the filtrate acidified with sulphuric acid, brought into a porcelain basin, and titrated cold with copper sulphate, using spots of 1 per cent. ferric chloride as indicator. The copper sulphate should contain 12·48 grammes of crystals per litre, and is equivalent to 0·01056 gramme of potassium ferrocyanide, or to 0·0072 gramme of Prussian blue, per 1 c.c. The process gives results which agree well with those of Knublauch's method (of which it is a modification). Drehschmidt's method shows higher figures, as it returns certain cyanogen compounds which are not ferrocyanides, and which do not yield Prussian blue. F. H. L.

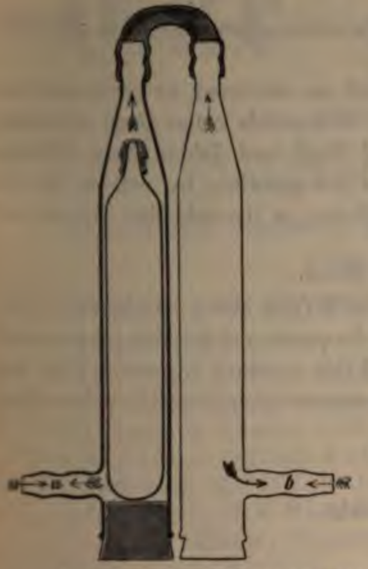
Estimation of "Available" Constituents in Soil. B. Sjollema. (*Chem. Zeit.*, 1901, xxv., 311.)—Processes like that of Dyer for the estimation of available phosphoric acid in soil, which are based on the extraction of a given weight of the sample for a prescribed time with a given quantity of some solvent, depend for their utility on the assumption that the whole of (that portion of) the constituent which is actually soluble in the liquid employed dissolves during the operation. This, however, is not correct; the speed of dissolution falls off rapidly, and when a state of equilibrium has been reached, there is no proof that all the available matter has been recovered. Sjollema quotes the results obtained on two soils, which show this: One was a light clay, that yielded 0·092 per cent. of phosphoric acid to boiling 12·8 per cent. nitric acid, this being presumably almost, if not quite, the whole of the phosphorus. When 50 grammes were treated by the Dyer process, 0·019 per cent. of phosphoric acid was obtained; and when the same sample was extracted again with citric acid, another 0·0185 per cent. was recovered. By treating the same soil with small separate quantities of solvent, allowing, say, 75 c.c. to act upon 50 grammes for a few hours with occasional agitation, running off the liquid, rinsing with 25 c.c., then soaking with 50 c.c., rinsing again, and so on, 0·0375 per cent. of phosphoric acid was eventually dissolved; and by percolation of 20 grammes of the soil in a narrow tube for seven days with 1 per cent. citric acid, the yield of phosphoric acid was 0·034 per cent., the total quantity of solvent used being about the same as in Dyer's process. Dyer's process thus returned only half the phosphoric acid really soluble in his reagent. The second specimen, a sandy loam, gave 0·212 per cent. to boiling nitric acid. Dyer's process showed 0·0528 per cent., and on further treatment with his solvent 0·015 per cent. more. Extraction with small quantities of solvent as above gave 0·0724 per cent. Here Dyer's process only returned about 73 per cent. of the phosphoric acid really soluble in 1 per cent. citric acid solution.

The author does not express any opinion as to whether 1 per cent. citric acid gives results of practical value, but he remarks that by his modification all the phosphoric acid which should (*ex hypothesi*) dissolve does so; and, moreover, in a soil containing much chalk, there is no need to add a larger volume of reagent at first, the extraction being simply repeated a greater number of times. The quantities mentioned above are intended only as suggestions; a larger volume can be used for each extraction, and the rinsing each time may be omitted. F. H. L.

The Use of Metallic Sodium in Blowpipe Analysis. Charles Lathrop Parsons. (*Journ. Am. Chem. Soc.*, xxiii., 159.)—That sodium may with great advantage be substituted for sodium carbonate in assisting the reducing action of the blowpipe flame has been pointed out by W. Hempel (*Zeits. anorg. Chem.*, xvi., 22), who, however, recommends certain details of manupulation which appear to be quite unnecessary. The reduction may be very simply carried out by flattening a piece of sodium 3 to 4 millimetres in diameter on a smooth surface, spreading the substance to be examined on it, and, after pressing it into the metal, kneading the whole into a ball with a knife. The metal should not be touched with the fingers, as the reaction sometimes commences spontaneously. The ball of sodium is placed on a depression in a block of charcoal and lighted with a match or flame, when a flash ensues, and reduction occurs. On heating the residue in the reducing flame, the sodium compounds sink into the charcoal leaving the metallic particles, which are tested as usual. Volatile metals yield their characteristic incrustations very readily, and the hepar reaction is easily obtained from the charcoal if sulphur was present. The method yields good results with most minerals, even chromite being readily reduced.

A. G. L.

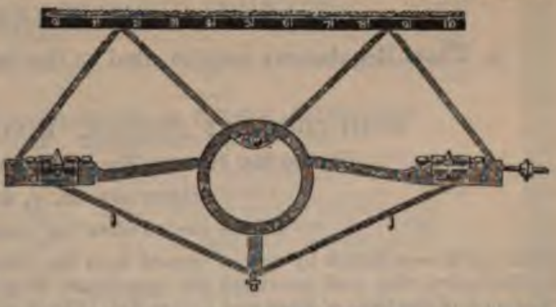
Rough Valuation of Strong or Fuming Sulphuric Acid. H. Rabe. (*Chem. Zeit.*, 1901, xxv., 345.)—This is a simple method of estimating approximately the strength of sulphuric acid which is suitable for use by workmen, etc. It depends on the fact that when a "fuming" acid is gradually diluted with ordinary acid containing less than 100 per cent. of hydrogen sulphate, the mixture continues to yield visible fumes on stirring until the excess of water in the weaker acid combines with the excess of anhydride in the strong acid, and the whole liquid consists of H_2SO_4. The only laboratory reagent needed is a chemically standardized sample of ordinary acid, if ordinary acid has to be valued, or of fuming acid, if fuming acid is to be examined, which should have roughly the same degree of concentration as the specimen under investigation. This reagent is employed to determine the fuming (or the fume-absorbing) power of a sample of fuming (weak) acid; and then by adding from a burette or pipette the specimen to be tested to the latter acid, the excess of SO_3 or of H_2O in the works sample can be calculated. Titration is performed in a beaker, stirring the liquid after each addition and blowing upon it; artificial cooling is not generally required. Foreign matter, dissolved or suspended, does not affect the results. F. H. L.



APPARATUS.

A Glass Gas Trap. K. Scholvein. (*Chem. Zeit.*, 1901, xxv., 397.)—This device is somewhat similar to the "steam trap" of the factory which separates steam from condensed water. There is a clear passage through the apparatus in either direction for any gas; but if liquid obtains access to either of the tubes it lifts the float contained in it, and eventually causes its capped end to make a tight joint against the conical top of the outer vessel. In the sketch the right-hand tube is shown in elevation; it also contains a float resembling that in the left tube. F. H. L.

A Balance of Constant Delicacy. (*Chem. Zeit.*, 1901, xxv., 294.)—The annexed diagram shows the construction of the beam of a balance, which is being patented by G. Hartner, of Ebingen, Würtemberg. It differs fundamentally from those in general use by having the upper pair of braces fastened to the ends of the beam outside the pan knife edges. This alteration prevents the gradual distortion of the beam with increasing loads, and renders it absolutely rigid; so that the relative positions of the centre of gravity and the centre of oscillation remain the same as when the balance is swinging empty. One of these balances designed to carry 200 grammes, was loaded with increasing weights up to 800 grammes in each pan; and yet, with an excess of 1 milligramme, always swung 15 divisions on the scale from zero. F. H. L.



SALE OF MILK REGULATIONS, 1901.

The Board of Agriculture, in exercise of the powers conferred on them by Section 4 of the Sale of Food and Drugs Act, 1899, have made the following Regulations:

Milk.

1. Where a sample of milk (not being milk sold as skimmed, or separated, or condensed, milk) contains less than 3 per cent. of milk-fat, it shall be presumed for the purposes of the Sale of Food and Drugs Acts, 1875 to 1899, until the contrary is

proved, that the milk is not genuine, by reason of the abstraction therefrom of milk-fat, or the addition thereto of water.

2. Where a sample of milk (not being milk sold as skimmed, or separated, or condensed, milk) contains less than 8·5 per cent. of milk-solids other than milk-fat, it shall be presumed for the purposes of the Sale of Food and Drugs Acts, 1875 to 1899, until the contrary is proved, that the milk is not genuine, by reason of the abstraction therefrom of milk-solids other than milk-fat, or the addition thereto of water.

SKIMMED OR SEPARATED MILK.

3. Where a sample of skimmed or separated milk (not being condensed milk) contains less than 9 per cent. of milk-solids, it shall be presumed for the purposes of the Sale of Food and Drugs Acts, 1875 to 1899, until the contrary is proved, that the milk is not genuine, by reason of the abstraction therefrom of milk-solids other than milk-fat, or the addition thereto of water.

EXTENT.

4. These Regulations shall extend to Great Britain.

COMMENCEMENT.

5. These Regulations shall come into operation on the First day of September, One thousand nine hundred and one.

SHORT TITLE.

6. These Regulations may be cited as the Sale of Milk Regulations, 1901.

HIGH COURT OF JUSTICE. KING'S BENCH DIVISION.

(Before Mr. JUSTICE WILLS and Mr. JUSTICE KENNEDY.)

PEARKS AND CO. *v.* KNIGHT.

(*From the " Times " of August* 10, 1901.)

THIS was a case stated by way of appeal from the Stipendiary Magistrate sitting at Longton in Staffordshire, who had convicted the appellants, Messrs. Pearks and Co., of an offence under Section 6 of the Sale of Food and Drugs Act, 1875, in selling butter not of the nature, substance, and quality demanded by the purchaser.

Mr. JOSEPH WALTON, K.C. (Mr. Avory, K.C., and Mr. H. D. Bonsey with him), for the appellants, said that the charge was that after the butter had been made milk was added in order to increase the weight of the butter by reason of the water contained in the milk. In the Margarine Act, 1888, butter was defined as being made of milk or cream. In the present case, that which was sold was not made of anything else. A process of making butter had been adopted which had the effect of making butter with more water in it than if it had not been adopted. There was no standard, and, therefore, so long as the article was made of milk there was no offence. There was nothing in the Act directed against any particular process of manufacture.

The COURT, without calling on Mr. Danckwerts and Mr. B. C. Brough, for the respondent, dismissed the appeal.

Mr. JUSTICE WILLS said that the purchaser asked for butter and he got that which had been butter, but by some ingenious process, the nature of which was probably a trade secret, milk was taken in considerable quantities and introduced into the butter in a state in which it was itself not butter. If it were subjected to the ordinary process by which butter was made and the natural amount of butter were turned out of it the object of the process would not be attained—namely, that some of the watery part of the milk, which would by the natural process of making the butter be turned out of it, should be left in the resulting product. That would not be done unless for the purpose of making a spurious profit. This was a right conviction, and the appeal must be dismissed with costs.

Mr. JUSTICE KENNEDY concurred.

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THE ANALYST.

OCTOBER, 1901.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

ARSENIC IN COAL AND COKE.

By Alfred C. Chapman, F.I.C.

(*Read at the Meeting, June* 5, 1901.)

wing to the necessity of obtaining malting fuel as free as possible from arsenic, the etection and estimation of that element in coal and coke has now become in many boratories a matter of almost everyday procedure. Up to the present time different ethods have been adopted by various chemists, and the somewhat serious dis-repancies which have frequently characterized the results are in themselves sufficient vidence that those methods are not altogether beyond criticism. Many analysts ave resorted to processes involving the more or less drastic treatment of the fuel ith sulphuric acid or nitric acid, or with mixtures of those acids under varying onditions. Others have adopted the method of deflagrating with an alkaline xidizing mixture, whilst others again have endeavoured to obtain the arsenic by istillation with hydrochloric acid and a reducing agent. For some time I adopted hat may be described as an acid extraction method, always, however, with a feeling hat it was open to considerable objection. When it became recognised that a considerable proportion of the arsenic in fuel remains in the ash when the fuel is burned, I made a number of determinations of this fixed arsenic, and found that in many cases the results were in excess of those which I had got by the acid process in my endeavour to obtain the total arsenic. It was obvious, therefore, that some other method was required, and after many experiments the process described below was devised. There can be little doubt that the arsenic in coal is present as arsenical pyrites, and that little exists in any other form.

I thought, therefore, that it might be interesting to ascertain how arsenical pyrites itself behaves when strongly heated in the presence of an excess of oxygen. Two samples of pyrites, "A" and "B," having respectively the following composition, were experimented with:

Sample.	Arsenic.	Iron.	Sulphur.
"A" ...	42·32 per cent. ...	33·66 per cent. ...	17·68 per cent.
"B" ...	44·27 " ...	34·65 " ...	17·34 "

Weighed quantities of these were heated to a bright red heat in a porcelain boat placed in a piece of ordinary glass-combustion tubing, the ignition being continued until the weight became constant, and the arsenic left in the residue (apparently as ferric arsenate) was then determined. In the case of "A" this amounted to 8·0 per cent., and in "B" 6·34 per cent. It will be seen, therefore, that a considerable proportion of the arsenic is incapable under these conditions of volatilizing.

When the pyrites was ignited with three times its weight of powdered wood-charcoal, the amount of arsenic remaining in the residue was increased, which may possibly be due to the fact that, owing to the dilution of the pyrites, the heating would necessarily be more gradual, and so increased opportunity for the oxidation of the arsenide would occur.

Before the method described below was arrived at, attempts were made to effect the complete oxidation of the coal by means of boiling concentrated sulphuric acid, as in Kjeldahl's nitrogen process, but so much difficulty was experienced in preventing the extraction of traces of arsenic from the glass that the method was soon given up. It was open, too, to the objection that anthracite coal requires a very considerable time for its oxidation, whilst coke is still more resistant. The method referred to at the commencement of this paper, and which I have now adopted, consists in the gentle ignition of the fuel with a mixture of magnesia and carbonate of soda, and is carried out in the following manner:

A weighed quantity of finely-ground fuel, depending upon the amount of arsenic it contains (usually from 0·5 gramme to 2 grammes will be found to be sufficient), is intimately mixed with about 2 grammes of pure calcined magnesia and ½ gramme of dry sodium carbonate in a deep platinum or, preferably, a silver crucible. This is then supported in a slanting position, and heated by a flame which is just capable of keeping the bottom of the crucible at a dull-red heat, the contents being stirred by means of a platinum wire from time to time. At the end of about one hour the oxidation will be complete, and ½ gramme of pure ammonium nitrate is then added, and the crucible ignited somewhat more strongly for about five minutes. This latter treatment is intended to oxidize any traces of sulphides which might have been formed, and which would, of course, tend subsequently to form insoluble arsenious sulphide, and so to remove it from the sphere of action in the Marsh apparatus. I have no actual proof that this part of the process is necessary, but I think it safer to adopt it. In this way, if care be taken not to heat the contents of the crucible too strongly, a powdery mass will remain, which can be readily removed. This is washed out into a beaker with dilute sulphuric acid, and the solution is then concentrated so as to decompose any nitrate that might have remained after the above ignition. This solution is then submitted to the modified Marsh-Berzelius method, and the mirror or mirrors so obtained are compared with standards. Should the fuel under examination be found to contain an unusually large amount of arsenic, it is obvious that the sulphuric acid solution may be made up to a definite volume and an aliquot portion used in the Marsh apparatus.

The following results will show that this method is accurate with very much larger amounts of arsenic than would ever occur in coal or coke.

In these experiments weighed quantities of arsenical pyrites of known composition were mixed with coal in which the amount of arsenic present was entirely negligible in comparison with that which was added, and the mixture treated as described above, save that the arsenic was determined gravimetrically as sulphide :

	Taken.	Found.
Arsenic	0·0050 gramme ...	0·0042 gramme
,, ...	0·0092 ,, ...	0·0096 ,,
,, ...	0·0096 ,, ...	0·0085 ,,
,, ...	0·0063 ,, ...	0·0055 ,,

In the following table numbers are given showing the amounts of arsenic present in six samples of coal, as well as in the coke made on a large scale from these samples, and in the ash, whilst the percentages of ash, of iron, and of calcium, are also given. It may be mentioned that these samples were gas coals, and were selected on account of the appreciable amount of arsenic which they contain :

Sample of Coal.	Arsenic in Coal, Grs. per lb.	Arsenic in Coke, Grs. per lb.	Ash of Coal, per cent.	Arsenic in Ash, Grs. per lb., calculated on the Coal.	Iron in Ash, per cent., calculated on Coal.	Calcium in Ash, per cent., calculated on Coal.	Alkalinity of Ash.
1	1·4	1·7	6·4	0·6	2·27	Trace	Distinct
2	0·5	0·7	16·2	0·3	1·71	Trace	Slight
3	0·7	1·0	6·7	0·3	2·62	None	Neutral
4	0·7	0·6	2·1	0·6	0·73	None	Neutral
5	0·9	1·1	12·5	0·7	1·96	None	Neutral
6	0·8	0·6	12·2	0·3	3·50	1·8	Very distinct

It will be seen that there is no definite relationship between the amount of arsenic present in the coal and that left in the ash on ignition, the proportion being apparently dependent upon the composition of the ash, as well as upon the manner in which the ignition has been performed. There can, I think, be little doubt, as I have stated above, that the arsenic in coal exists almost entirely as a constituent of the pyrites present, and that little exists in any other form. The two following experiments are of interest in this connection. From one of the two samples of coal referred to in the above table the pyritic portions were picked out. An estimation of the arsenic present in this sample showed no less than 1·9 grains per pound, whilst only 0·2 grain per pound was present in the non-pyritic portion. In the second experiment, two samples of New Zealand coal, taken from different parts of the same seam, and giving on analysis the following results, were examined :

	No. 1.	No. 2.
Ash	4·55 per cent. ...	1·60 per cent.
Volatile sulphur ...	2·46 ,, ...	3·22 ,,
Total arsenic ...	0·2 grain per pound ...	0·5 grain per pound
Arsenic in ash ...	0·1 ,, ,, ...	0·18 ,, ,,

Although No. 2 contained much less ash than No. 1, yet in the former there was a distinct quantity of iron, whilst the latter was practically free from that element, and it will be seen that the amount of arsenic in No. 2 was distinctly greater than in No. 1. As examples of the application of this method to higher class fuels than those referred to above, the following numbers obtained with Welsh anthracites of high quality may be recorded :

	No. 1.	No. 2.
Total arsenic ...	0·03 grain per pound ...	0·035 grain per pound
Arsenic in ash ...	0·01 ,, ,, ...	0·020 ,, ,,

That the method described in this paper is capable of yielding approximately accurate results in the analysis of fuel admits of no doubt, and it has the merit of being easily performed, and of involving the use of materials which can without difficulty be obtained perfectly free from arsenic. It is obvious that it might with slight modification be employed in the analysis of other organic materials than coal and coke.

DISCUSSION.

Dr. MORRIS said that he was inclined to attach more importance than Mr. Chapman to the total arsenic in fuel, for his own experience appeared to indicate that the influence of any arsenic in the ash or dust which accumulated in the kiln on the quantity of arsenic subsequently found in the malt was as great as, or greater than, the influence of the arsenic actually volatilized during kilning. Of course, from the very nature of the contamination in the fuel a serious difficulty existed in connection with sampling. The arsenical pyrites, from which the arsenic was derived, occurred in patches or pockets in the fuel, so that while a sample from one portion of a particular truck-load might be perfectly free from arsenic, a sample from another portion of the same truck-load might be very highly contaminated with arsenic. For that reason he thought it advisable to use for analysis as large a quantity as possible of the fuel, and he thought that the quantity recommended by Mr. Chapman would be far too small to insure anything like a fair average sample of the fuel, while any slight impurity in the reagents used would have a greater percentage effect on the ultimate result than if a larger quantity were worked with. Even working with 50 grammes he had found it difficult to get a really fair sample. The process of analysis he had adopted was practically that which Mr. Chapman had mentioned in the first instance—viz., digestion of the finely-powdered coke or coal with hydrochloric acid to decompose sulphides, and then with fuming nitric acid and sulphuric acid in order to oxidize as far as possible the organic matter, the residue being washed and the extract "Marshed." Even if the whole of the arsenic were not obtained in this way, the figures obtained were comparative, and after all that was what was really wanted. The structure of the kiln had a very great influence with respect to the presence or absence of arsenic in the malt. The whole question was really one which was hardly capable of being dealt with by ordinary methods of analysis, as so many other factors had to be taken into consideration. The question had recently been raised whether arsenic had not always been present to a greater or less extent in beer. In this connection, he might state that in three samples of old beer which he had recently examined (one brewed in 1798, one in 1875, and one in 1886) he had found respectively approximately $\frac{1}{50}$, $\frac{1}{80}$, and $\frac{1}{75}$ of a grain of arsenic per gallon. These were strong beers, the original gravity ranging from 1090 to 1112. In the case of the two more recent samples he knew that the malt used had been cured with a mixture of anthracite and coke. The first sample was probably brewed before the introduction of coke, but whether the fuel used in that case in drying and curing the malt was coal alone or whether it was a mixture of coal and wood he did not know.

Mr. Fairley said he did not quite agree with Mr. Chapman in trying to apply to the case of coal and coke the inferences drawn from analyses of what might be called pure arsenical pyrites. He had looked up the analyses of a great many samples of pyrites used in the manufacture of sulphuric acid, which might be assumed to be similar in character to the pyrites present in coal, and he had found that very often the percentage of arsenic was approximately 1 per cent. of that of sulphur. Mr. Chapman had referred to the possible influence of different basic constituents in fixing the arsenic. In his own opinion, the substance most likely to fix the arsenic was ferric oxide, which was capable of both reduction and oxidation. In the case of pyrites in which the arsenic formed only a very small proportion of the total pyrites, a larger proportion of the arsenic would be fixed than in cases in which the quantity of arsenic was greater. In regard to coke, he had been in the habit of assuming—though he did not know that he had very good evidence for it—that about one-half of the arsenic he could find was volatile. A good many of Mr. Chapman's figures appeared to agree with this, but it was obviously only a very rough assumption. He was quite certain that Mr. Chapman's method of determining the total arsenic was better than the acid method which had hitherto been used, and which he had always thought could only yield a certain proportion of the total arsenic, and it seemed from the results now obtained that that proportion was not really very much greater than the actual proportion of volatile arsenic. Of course, as Dr. Morris had pointed out, the arsenic carried off in the dust (the quantity of which dust would vary with the strength of the current of gases circulating through the kiln) did not consist merely of volatile arsenic, and might even be arsenic present in some of the ash itself carried off in a finely-divided state. His own recent experiments showed that in kilns of very different construction, made according to what might be called the newest methods and also according to the oldest methods, the dust which collected on the under side of the tiles actually contained as much as 1 per cent. of total arsenic.

Mr. Hehner desired to ask Mr. Chapman what proportion of carbonate of lime he had used in his attempts to retain the arsenic by this means.

Mr. Chapman said that about equal quantities of arsenical pyrites and of carbonate of lime were used.

Mr. Hehner said that in a process which had been recently patented the use of lime was proposed for retaining the arsenic. It might be said that as the coal burned the lime became converted into carbonate; but when in practical analytical work the coal was mixed intimately with calcium oxide and heated to a red heat there was very little absorption of carbonic acid by the lime, and it might be taken that the action of actual lime would be different from that of calcium carbonate.

Mr. Chapman said that he had used calcium carbonate because it probably existed in that form in the coal.

Mr. HEHNER said it was obvious that some alkaline medium must be used to retain the arsenic, because there were no vessels available by the use of which the complete absence of arsenic could be insured, and some method like that suggested by Mr. Chapman was a necessity. He (Mr. Hehner) was astonished at the very large quantities of volatile arsenic that Mr. Chapman had found in some of the samples of coal referred to. When it was considered that between 5 and 6 pounds of malt were prepared on the average by about 1 pound of coal, it would follow that, altogether disregarding the ash, malt prepared with coal like Mr. Chapman's sample No. 1, which contained 0·8 grain of volatile arsenic per pound, must contain at least 0·16 grain of arsenic per pound.

Mr. CHAPMAN observed that these coals were all gas coals, such as would never be used for malting purposes.

Mr. HEHNER, continuing, said that, in spite of what Dr. Morris had said, he thought it important to distinguish between total and volatile arsenic. It was possible, by proper construction of the malt-kilns, to prevent the access to the malt of any considerable proportion of ash, and, as matters at present stood, failure to prevent it by proper construction amounted to culpable negligence. But there was no known method at present of preventing access of volatile arsenic, and it seemed very hard to reject a fuel which might contain, comparatively speaking, a large quantity of total arsenic when practically none of that arsenic might be volatile. He had made a number of experiments, and had found that in most cases the proportion of volatile to total arsenic was far less than one-half. He thought that the present ideas as to the volatility of arsenic would have to be altered. If the question were asked, What happened when an arsenical body was mixed with 90 per cent. of carbon? the reply would be that the arsenic was reduced and volatilized; but this was not actually the case, and some attempt ought seriously to be made to ascertain in what form the arsenic existed. He understood that Mr. Chapman's endeavours to connect it with the iron or any other of the ash constituents had failed. It seemed to him that it must be in a state of organic combination, and that it burned away just as in the case of sulphur, which in coal was undoubtedly partly in a state of organic combination. In endeavouring to retain the arsenic by means of lime, he had found that the lime used was itself not inconsiderably contaminated with arsenic and on heating some white marble chips in a platinum crucible to red heat and afterwards in a blowpipe-flame, he had found that that again was contaminated with arsenic.

Dr. MORRIS suggested that, having regard to the temperature below the kiln tiles and to that at which arsenic trioxide was volatilized, the bulk of the volatile arsenic would probably cease to exist in a state of vapour and be precipitated with the dust, so that really the question of dust was the chief one to be dealt with in regard to contamination.

Mr. BAKER thought that, in regard particularly to what Dr. Morris had said an to the way in which malt-kilns were very largely constructed in this country, it woul not be of much assistance to return separately the amounts of volatile and of fixe arsenic—at any rate until the present knowledge of the subject was increased. H also had tried to retain the arsenic by means of lime, but found that the lin contained about $\frac{1}{50}$ of a grain of arsenious acid per pound, a similar quantity being found after the same lime had been ignited to bright red heat for an hour in a muffle, showing that, up to that point at any rate, the arsenic in the lime was fixed. Certainly, so far as malting fuels were concerned, treatment with certain proportions of lime did seem to hold back the arsenic. This he had been able to test on a practical scale with very satisfactory results.

The PRESIDENT (Dr. Voelcker) said that the main point upon which information was wanted seemed to be that referred to by Mr. Hehner—namely, the form in which the arsenic existed, and whether or not the volatile and non-volatile portions were equally harmful. The fact, which had now been so strongly brought out in the case of arsenic, that a considerable proportion remained behind in the ash, was also common to other bodies than arsenic. For instance, if a very highly organic body containing a certain proportion of nitrogen were charred to the state of charcoal, it would seem at first sight that there ought to be no nitrogen left in the charcoal; an appreciable quantity of nitrogen would, however, be found, even though the charring process had been thoroughly carried out. The question seemed somewhat on a par with that of the fertilizing constituents in a soil, which might be partly in an available condition and partly in a non-available or insoluble condition.

Mr. CHAPMAN said that he fully agreed that it was very important to know the proportion of total arsenic as well as that of volatile arsenic, and for this reason he thought that some such process as he had described, which gave both the total and the volatile arsenic, was an advantage. He agreed with Dr. Morris as to the importance of accurate sampling, but he did not agree at all that it was necessary to work on a very much larger quantity than 0·25 or 0·50 gramme. In his own practice he recommended that samples of coal for analysis should consist of about 3 or 4 pounds, representing a much larger and carefully-drawn sample. A thoroughly representative portion was then prepared, from which the small quantity ultimately required for analysis was drawn. It therefore would not matter whether the quantity actually analysed was 0·5 gramme or 50 grammes. In most of these cases, even working upon the small quantities recommended, the mirrors obtained were as dark as could possibly be used for purposes of comparison, 0·25 gramme in some cases yielding a mirror of a depth beyond which it was not possible to go. Even in the case of good anthracite, 1 gramme yielded a mirror as distinct as it was advisable to use for comparison. He must also dissent from Dr. Morris in regard to the application of the "acid method." He had used such a method himself until recently, but regretted having done so after comparing its results with those of the present method, which he knew to be much more accurate. In regard to the question of impurities in the materials, he had found it possible—though only after many experiments and much trouble—to obtain materials which gave what might be termed "perfect blanks" under the conditions of the experiment for half an hour. Any minute trace of impurity that might possibly be present could not have any appreciable influence in cases like those now under consideration, in which the quantities of arsenic were so much greater than those given by average malts, containing, say, from the $\frac{1}{100}$ to the $\frac{1}{200}$ of a grain of arsenic per pound. As a matter of fact, it would be quite impossible to obtain anything like the total quantity of arsenic from such large quantities as Dr. Morris mentioned. Mr. Fairley had suggested that inferences ought not to be drawn, from experiments with pyrites, in regard to the form in which the arsenic existed in the coal, and he (Mr. Chapman) was sorry to say that, as a matter of fact, it had not been possible to draw any such definite conclusions. He wanted, however, in the first place to see how a substance like pyrites would behave when ignited in the presence of oxygen, and it was interesting to find that about an eighth of the arsenic remained behind in the ash, existing almost certainly as arsenate of iron. He agreed with Mr. Fairley that it was exceedingly probable that the arsenic was fixed chiefly by the iron, and this seemed to be shown by his own experiments with carbonate of lime. In fact, he had never found any appreciable quantity of arsenic in any coal the ash of which contained no iron. He might perhaps repeat that the coals used in these experiments were gas coals and not malting coals. His idea had been that such coals would give larger results, which would possibly have a clearer meaning than the smaller numbers yielded by purer samples.

OIL OF CITRON.

BY HERBERT E. BURGESS.

(*Read at the Meeting, May* 1, 1901.)

OWING to the extensive adulteration of citron oil, considerable doubt attaches to many of the published numbers representing its physical and chemical constants. A sample of the oil of undoubted genuineness having been placed at my disposal, it occurred to me that the results which I have obtained on its examination might possibly be of interest and use to the members of the Society.

There has always been considerable confusion between lemon and citron oil, as in France and Sicily lemon oil is known as "Essence de Citron," while citron is known as "Essence de Cedrat."

Citron oil is stated to be expressed from the rind of *Citrus medica risso*; but, in fact, a smaller variety of the fruit is used, the true citron fruit being used almost exclusively for the production of candied peel.

It is generally known that the majority of citron oil sent to this country is nothing more than a mixture of lemon oil with verbena, and, in some cases, a small quantity of otto of roses is used as a sweetening agent.

The oil referred to above gave on examination the following constants:

Specific gravity, 15° C., 0·8513
Refractive index, $[N]_D$, 20° C., 1·4750
Optical rotation, $[\alpha]_D$, 100 mm. + 80° 13′

On distilling the oil at a pressure of 10 millimetres, the following fractions were obtained:

1. 60–62°	12 per cent.	rotation + 86° 30′
2. 62–64°	80 ,,	rotation + 85° 30′
3. 64–85°	5 ,,	rotation + 13° 30′

Numbers 1 and 2 were refractionated and distilled several times over clean sodium to remove any oxygenated constituents. The main fraction thus obtained was identified as limonene, boiling at 173° to 174° C. at atmospheric pressure, and having a rotation of +89°. Possibly with this fraction there was a small percentage of the dipentene, which would account for the rotation being somewhat lower than is generally given for limonene, but I was unable to make any further separation.

The third fraction was examined for citral by Tiemann's cyanacetic acid reagent, and I obtained the well-defined citralidene-cyanacetic acid, having a melting-point of 120° to 121° C.

With the mercuric sulphate reagent, as described in the ANALYST, vol. xxv., p. 265, it gave the blood-red coloration peculiar to citral.

A determination of the citral in the original oil gave the following figures:

By sodium bisulphite method (i.) 6·2 per cent.; (ii.) 5·8 per cent.
By hydroxylamine method, 5·7 per cent.

The original oil had a considerable deposit of resinous and crystalline matter, which was filtered off and extracted with chloroform; on standing crystals were formed which were filtered off and dissolved in petroleum spirit to free them from resinous matter. The crystals thus obtained were finally recrystallized from alcohol. Well-formed needle-shaped crystals were obtained, having a melting-point of 145° C., with a marked blue fluorescence in alcoholic solution. The crystals were soluble in alcoholic potash and reprecipitated by acids; quite neutral to litmus; slightly soluble in boiling water, from which they crystallized out on cooling; soluble in strong sulphuric acid. Boiling Fehling's solution was not reduced by them.

Two combustions gave:

	I.	II.
Carbon	64·77	64·88
Hydrogen	5·40	5·24
Oxygen	29·83	29·88

Molecular weight determination gave 355, corresponding with a formula: $C_{18}H_{18}0_6$.

I believe this substance to be identical with that observed by Crismer, and more recently by Theulier.

The following table gives my results and those of other workers.

	Specific Gravity.	Rotation.	Boiling-point.	Citral.	Refractive Index.
Sample A ...	0·8513	+80° 13′	60°–100° (10 mm.)	6 %	1·4750
Sample B ...	0·8568	+70° 13′	58°– 90° (10 mm.)	5 %	1·4755
Gildemeister...	0·8710	+67° 8′	177°–220°		
Charabot ...	0·8700	+67° 0′	177°–220°		
Parry... ...	0·86–0·87	+66°–76°			

Sample A represents the genuine oil spoken of above.

Sample B was an oil obtained from another source, and guaranteed to be pure, but on examination it was found to be adulterated with a considerable quantity of lemon oil.

It will be seen at once, by referring to the above table, that the physical constants of a genuine citron oil are widely different from the oils examined by different workers.

I understand that citron oil is only expressed in Sicily to special order.

DISCUSSION.

Dr. MORRIS asked if the white, needle-shaped crystals referred to were found in the oil itself.

Mr. BURGESS said that the oil, as he received it, contained a considerable deposit, which had been filtered off, and from this the crystals were extracted.

Dr. MORRIS said that some years previously (*Journ. Chem. Soc.*, 1882), in the course of an investigation in connection with the hydrocarbons occurring in resin spirit, he had found in certain samples crystalline needles which behaved with acid similarly to those described by Mr. Burgess, and which he had identified as heptene glycol.

Mr. CHAPMAN said that an important point in connection with this sample was the fact that its rotation was, he believed, some 10° or 12° higher than the highest recorded number for citron oil. The boiling-point of the sample was very much lower than those recorded by Gildemeister and one or two other observers, though it was somewhat doubtful whether any real comparison could be made with these figures, because they represented merely the boiling-points between which the main fraction distilled, whereas those of Mr. Burgess represented the total range from minimum to maximum.

Mr. BURGESS said that he had never observed in lemon oil a rotation of over 67°, whereas in this particular oil it was over 80°, or 10° higher than the highest recorded for lemon oil. The specific gravity, of course, was very much lower. The references to the range of boiling-point in the different works on the subject did not indicate at what pressure the boiling-points were determined, but it was probably at atmospheric pressure. The whole range from beginning to end would be taken. Any one particular fraction would have a much smaller range.

THE USE OF PARTIALLY STERILIZED MILK CULTURES IN JUDGING THE PURITY OF WATER.

BY H. DROOP RICHMOND, F.I.C.

(*Read at the Meeting, June* 5, 1901.)

KLEIN has recommended a search for *B. sporogenes enteritidis* as an indication of the purity of water; this is done by inoculating 10 c.c. of sterile milk with 1 c.c. of the water, heating to 80° C. for fifteen to twenty minutes, and incubating anaerobically at 37° C.; a curdling of the casein in dry-looking masses, with enormous evolution of gas, together with a peculiar odour, is characteristic of this organism.

For the past few months I have always made this test, and have also made a second milk-cultivation by inoculating 10 c.c. of sterile milk with 1 c.c. of water, heating to 50° C. for ten minutes, and incubating aerobically at 37° C. I find that it takes two and a half minutes to heat up 10 c.c. of milk and 1 c.c. of water to the temperature of the bath, and therefore plunge the tubes in cold, and keep them there for twelve and a half, seventeen and a half, or twenty-two and a half minutes, according as I wish to heat ten, fifteen or twenty minutes respectively. There is no difference in the results obtained by heating to 80° C. for fifteen or twenty minutes, and latterly I have always adopted fifteen minutes.

My results with eighty samples of water are given in the tables below. Table I. contains waters which from a careful examination of the source show no signs of pollution; none of these curdled milk heated to 50° C., and only one milk heated to 80° C. Table II. contains waters judged to be free from sewage pollution, but which are more or less exposed, and may be contaminated by decaying vegetable matter and small animals; the bulk of these curdled milks heated to 50° C. in less than three days, and the number of micro-organisms was usually high; chemically there is little to object to in these waters. Table III. contains waters known to be polluted; six out of twenty waters do not curdle milk, and four of these do not give a large number of micro-organisms on cultivation; two, in fact, which are perhaps the worst chemically, and in which the pollution is most plainly established by inspection, are nearly sterile. The other waters in this table all curdle milk heated to 50° within two days.

In Table IV. are classed the doubtful waters; these include waters which change in analysis in six months to an appreciable extent, waters from wells with unprotected sides, waters from springs rising from manured gathering ground, and finally waters about which I have no information.

In two cases, in which pollution was established where chemical examination did not show this, milk heated to 50° was curdled in thirty-six hours, and this was the first indication of pollution.

From a consideration of the results I am of opinion:

1. That any water curdling milk heated to 50° for ten minutes in two days or less is very suspicious.

2. That a water may be considered good if it does not curdle milk heated to 50° for ten minutes, and has a favourable chemical analysis.

3. That some badly polluted waters are bacteriologically good, and these do not curdle milk.

I think that the cultivation of milk heated to 80° has much less value than that heated to 50°, though doubtless the detection of *B. sporogenes enteritidis* would be conclusive; I have never found this organism in the waters I have examined.

DISCUSSION.

Mr. BAKER inquired whether the author had been able to differentiate between pathogenic and non-pathogenic organisms by this process. At certain times of the year milk was very apt to be contaminated with *Bacillus subtilis*, which formed spores that would not be destroyed by heating to 50° C.; and although this organism was perfectly harmless, he understood that if it happened to be present in the milk the results of this test would still be taken to indicate that there was ground for suspicion.

TABLE I.—

Source.	Microscopic Examination.	Chlorine.	Free Ammonia.	Albuminoid Ammonia.	N_2O_5	= Nitrogen.
Public supply	good	1·4	0·007	0·0055	trace	trace
Well ...	„	4·0	0·003	0·006	1·15	0·30
„ ...	„	1·2	0·002	0·002	1·5	0·39
„ ...	„	1·4	none	0·0025	1·35	0·35
„ ...	„	2·0	0·0145	0·0045	none	none
„ ...	„	2·0	0·0295	0·004	„	„
„ ...	„	1·3	0·001	0·0115	1·45	0·38
„ ...	„	2·05	none	0·012	1·1	0·29
„ ...	„	1·4	0·003	0·0045	none	none
„ ...	„	1·25	none	0·005	1·3	0·36
Public supply	„	2·3	„	0·0025	none	none
Well ...	„	6·4	0·071	0·0075	„	„
„ ...	„	1·8	none	0·007	4·0	1·04
„ ...	„	1·35	„	0·004	1·6	0·41
„ ...	„	1·7	„	0·005	2·8	0·73
„ ...	„	1·35	„	0·0035	1·7	0·44
Spring ...	„	1·2	„	0·0095	none	none

TABLE II.—GOOD

Source.	Microscopic Examination.	Chlorine.	Free Ammonia.	Albuminoid Ammonia.	N_2O_5	= Nitrogen.
Spring ...	good	1·3	0·006	0·0175	none	none
„ ...	—	—	—	—	—	—
„ ...	good	1·25	none	0·004	1·1	0·29
„ ...	„	1·3	„	0·005	1·2	0·31
„ ...	„	1·8	0·002	0·0095	none	none
„ ...	„	1·9	0·0035	0·0145	„	„
„ ...	„	1·55	0·003	0·003	1·9	0·49
„ ...	„	1·75	none	0·004	2·0	0·52
„ ...	bad	1·7	trace	0·0095	none	none
„ ...	good	0·7	„	0·012	trace	trace
„ ...	doubtful	1·2	none	0·008	0·4	0·10
„ ...	bad	1·25	0·004	0·007	0·85	0·22
„ ..	doubtful	1·2	0·0065	0·008	1·6	0·41
„ ...	good	2·2	0·0065	0·008	0·6	0·16
Reservoir	„	2·0	trace	0·0105	0·15	0·04

Mr. CHATTAWAY said that in applying this test he had met with one or two peculiar cases which might perhaps be accounted for in the way suggested by Mr. Baker; but, generally speaking, if coagulation took place, even if the chemical results pointed to the water being fairly good, he thought it worth while to inspect

GOOD WATERS.

Oxygen absorbed in 4 hours.	Gelatin Colonies per c.c.	Agar Colonies per c.c.	Milk heated to 50° C. for 10 minutes.	Milk heated to 80° C. for 15 to 20 minutes.	Remarks.
0·021	10	2	no change	no change	
0·015	50	none	,,	,,	
0·004	10	,,	,,	,,	Abyssinian tube well.
0·006	300	,,	,,	,,	
0·013	none	,,	,,	,,	deep well.
0·027	,,	,,	,,	,,	
0·036	10	,,	,,	,,	
0·036	90	,,	,,	,,	
0·023	none	,,	,,	,,	
0·027	,,	,,	,,	,,	
0·048	,,	,,	,,	,,	
0·038	Pure culture, *B. Aurantiacus.*	,,	,,	,,	deep well.
0·025	120	1	,,	,,	
0·008	40	none	,,	,,	
0·015	2,440	,,	,,	,,	
0·010	none	,,	,,	c. 3 days	
0·067	110	,,	,,	no change	

WATERS WHICH ARE EXPOSED.

Oxygen absorbed in 4 hours.	Gelatin Colonies per c.c.	Agar Colonies per c.c.	Milk heated to 50° C. for 10 minutes.	Milk heated to 80° C. for 15 to 20 minutes.	Remarks.
0·188	1,470	34	c. 36 hrs., gas	no change	gathering ground in wood.
—	very large	9	c. 2 days	,,	
0·017	40	none	no change	,,	travels a little distance exposed.
0·017	2,520	,,	c. 2 days	,,	
0·102	very large	55	,,	c. 2 days	two open tanks.
0·052	,,	141	c. 36 hrs., gas	no change	
0·017	540	2	c. 2 days, gas	,,	loosely-covered tank.
0·008	170	none	no change	,,	
0·096	large	21	c. 24 hrs., gas	c. 2 days	open reservoir.
0·092	48	14	c. 36 hrs., gas	no change	exposed at source.
0·036	40	28	,, ,,	c. 3 days	
0·030	large	1	c. 2½ days	no change	carted three miles in a cart.
0·084	10,000	12	c. 2 days	,,	
0·036	2,220	none	no change	,,	exposed at source.
0·040	320	,,	,,	,,	fed by springs.

the source of the water very carefully. The samples to which he had applied the test were from a district with which he was well acquainted, where the water had been examined very thoroughly, and where, moreover, he rarely analysed a samp without personally seeing the well or other source of supply.

Table III.—

Source.	Microscopic Examination.	Chlorine.	Free Ammonia.	Albuminoid Ammonia.	N_2O_3	=Nitrogen.
Well ...	good	12·95	0·003	0·0095	12·0	3·11
,, ...	fair	0·5	0·004	0·032	trace	trace
,, ...	bad	0·8	0·0165	0·020	none	none
,, ...	good	13·6	none	0·0135	6·7	1·74
,, ...	doubtful	12·9	,,	0·0115	5·4	1·40
,, ...	good	3·3	0·003	0·0105	4·9	1·27
,, ...	bad	13·3	0·002	0·016	27·0	7·00
,, ...	,,	4·9	0·0085	0·020	3·5	0·91
,, ...	doubtful	5·4	none	0·014	4·15	1·08
,, ...	bad	11·1	0·180	0·083	16·7	4·33
,, ...	good	3·55	0·028	0·028	7·5	1·94
,, ...	bad	2·3	none	0·008	1·5	0·39
,, ...	good	6·1	0·057	0·012	15·0	3·89
,, ...	doubtful	11·9	0·0025	0·0115	6·2	1·61
		11·9	0·001	0·0095	9·2	2·39
,, ...	bad	3·2	0·013	0·0085	none	none
,, ...	good	1·55	trace	0·004	1·7	0·44
,, ...	,,	13·8	0·0135	0·0195	6·7	1·74
,, ...	,,	3·05	none	0·006	4·0	1·04
,, ...	doubtful	1·5	0·002	0·004	3·3	0·86
,, ...	good	2·4	0·003	0·0035	7·1	1·84
,, ...	,,	3·55	0·0085	0·0145	7·7	2·0

Table IV.—

Source.	Microscopic Examination.	Chlorine.	Free Ammonia.	Albuminoid Ammonia.	N_2O_5	=Nitrogen.
Well ...	good	3·6	0·005	0·016	none	none
,, ...	,,	6·65	0·126	0·010	,,	,,
Public supply	,,	1·3	0·0085	0·011	,,	,,
Well ...	,,	1·1	none	0·0115	0·35	0·09
,, ...	doubtful	1·9	0·0185	0·070	1·5	0·39
,, ...	good	1·8	none	0·0035	3·7	0·96
,, ...	doubtful	1·6	none	0·0045	2·3	0·60
,, ...	good	1·9	0·0045	0·007	none	none
,, ...	bad	2·35	0·0045	0·0115	0·75	0·19
Public supply	good	3·5	0·009	0·006	1·6	0·41
,, ,,	,,	0·65	0·008	0·0025	none	none
Well ...	,,	4·9	0·005	0·0025	,,	,,
,, ...	,,	4·85	0·002	0·0025	,,	,,
,, ...	,,	2·7	0·002	0·004	,,	,,
,, ...	,,	3·15	0·004	0·004	0·85	0·22
Spring ...	,,	1 0	trace	0·004	3·0	0·78
,, ...	doubtful	1·3	none	0·0065	2·4	0·62
Rain-water	fair	0·5	trace	0·0085	none	none
Well ...	good	2·2	0·019	0·006	,,	,,
,, ...	doubtful	2·3	0·0095	0·006	,,	,,
Spring ...	good	1·3	0·0015	0·0045	3·7	0·96
Well ...	,,	2·3	none	0·009	3·0	0·78
,, ...	,,	1·3	0·003	0·0075	1·8	0·47
,, ...	,,	1·6	none	0·0055	2·4	0·62
,, ...	,,	1·4	0·001	0·006	1·3	0·36
,, ...	,,	1·95	0·0065	0·015	2·0	0·52
,, ...	,,	1·8	0·004	0·0075	2·5	0·65

Bad Waters.

Oxygen absorbed in 4 hours.	Gelatin Colonies per c.c.	Agar Colonies per c.c.	Milk heated to 50° C. for 10 minutes.	Milk heated to 80° C. for 15 to 20 minutes.	Remarks.
0·025	very large	15	c. 36 hrs., gas	c. 3 days	
0·097	" "	34	" "	c. 2 days	soft-water well, fed from roof; leaky.
0·107	260	large	c. 2 days	c. 3 days	
0·055	very large	124	c. 36 hrs.	"	half under house.
0·057	1,150	5	c. 2 days, gas	no change	
0·048	620	12	c. 36 hrs., gas	c. 2 days	
0·067	140	67	no change	no change	
0·043	—	—	"	"	
0·046	410	3	"	"	
0·202	170	large	c. 36 hrs.	"	
0·248	20	none	no change	"	
0·040	1,100	*B. coli* present	c. 36 hrs., gas	"	pollution established.
0·046	20	none	no change	"	well between privy, cess-pit, stables and yard.
0·050 0·046	very large	large	c. 36 hrs.	"	under house.
0·112	" "	"	c. 2 days	c. 3 days	drainage from yard entered through recent opening.
0·017	140	27	c. 2 days, gas	"	slight drainage from yard.
0·107	7,000	17	c. 36 hrs.	no change	
0·021	50	1	no change	"	
0·021	very large	11	c. 36 hrs., gas	"	
0·011	710	large	c. 2 days	"	
0·058	—	none	no change	"	

Doubtful Waters.

Oxygen absorbed in 4 hours.	Gelatin Colonies per c.c.	Agar Colonies per c.c.	Milk heated to 50° C. for 10 minutes.	Milk heated to 80° C. for 15 to 20 minutes.	Remarks.
0·024	130	none	no change	no change	gault waters; probably polluted and purified.
0·038	4,110	6	"	"	
0·134	1,340	1	"	"	
0·063	1,920	10	c. 3 days	"	situated at bottom of a hill; not protected.
0·258	690	3	no change	"	
0·027	60	none	"	"	
0·031	1,450	10	c. 3 days	"	
0·021	240	none	no change	"	sides unprotected from surface-water.
0·065	12,360	4	c. 3 days	"	
0·025	70	none	c. 2 days, gas	"	
0·188	260 B. Fl. Liq.	1	" "	"	
0·006	large	10	c. 36 hrs.	"	
0·002	340	1	c. 2 days	"	
0·010	20	1	c. 3 days	"	
0·010	40	4	c. 2 days	"	
0·025	360	7	no change	"	gathering-ground manured.
0·023	140	7	"	"	
0·038	90	9	c. 3 days, gas	c. 3 days	
0·009	2,470	1	no change	no change	
0·023	1,440	10	"	"	
0·118	very large	—	c. 2 days, gas	"	gathering-ground manured.
0·059	large	none	no change	"	
0·051	"	"	c. 2 days	"	surface-water finds access.
0·053	very large	large	no change	"	top of well not closed.
0·027	6,000	3	"	"	
0·082	1,780	none	c. 2 days	"	
0·041	very large	"	no change	"	

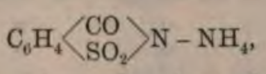
The PRESIDENT (Dr. Voelcker) remarked that milk was a substance which, as h
been pointed out, was from its very nature particularly liable to undergo changes
a kind that would be influenced by other conditions than those which might be d
to the water alone. Furthermore, difficulties would probably arise in a test such
this from the fact that a water might contain a certain quantity of dissolved orga
matter which would produce results apparently unfavourable, but which might
itself be of perfectly harmless origin.

Mr. RICHMOND said that the test did not admit of differentiation betwe
pathogenic and non-pathogenic organisms. Of course, if *Bacillus subtilis* we
present in the water it would affect the milk. In some cases the action on mi
yielded indications which could not be ignored. In one of the instances he h
mentioned the existence of the pollution was clearly confirmed by an inspection of t
well, though the chemical results were not conclusive.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

The Determination of Saccharin in Food-Products. H. Défournel. (*Jou
Pharm. Chim.*, 1901, xiii., 512-515.)—The author's method is based upon the fa
that saccharin forms a soluble ammoniacal salt on treatment with ammoni
hydroxide,



$$C_6H_4\left\langle\begin{matrix}CO\\SO_2\end{matrix}\right\rangle N-NH_4,$$

and that this salt, like all ammonium salts, gives off nitrogen on treatment with
alkaline solution of sodium hypobromite, without decomposition of the saccha
group. The amount of saccharin is then calculated from the volume of gas liberat

Two hundred and fifty c.c. of the liquid under examination are acidified with
phuric acid (1 : 10), and shaken in a separating funnel with three successive porti
of 50 c.c. of a mixture in equal parts of ether and petroleum spirit. The uni
extracts are washed with water and evaporated. The residue is treated with
monium hydroxide, and the excess of the latter evaporated on the water-bath.
ammonium salt is taken up with water and the nitrogen determined in a nitrome
as in the case of urea.

The volume of nitrogen in tenths of a c.c., divided by the factor 8·9, gives
weight of the saccharin in centigrammes.

The method gives good results when the amount of saccharin is not less t
0·03 gramme, but with smaller quantities it is advisable to concentrate the liq
containing it.

C. A. M

The Examination of Lemon-Juices. **E. Spaeth.** (*Zeit. für. Untersuch. der Nahr. und Genussmittel*, 1901, 529.)—For the purpose of discriminating between natural lemon-juice and that artificially prepared from citric or tartaric acid, determinations of the ash and the alkalinity of the ash are the most useful. Natural lemon-juice contains about 0·4 per cent. of ash, using about 6 c.c. per cent. of normal acid for neutralization, whilst citric acid solutions have no appreciable ash, with, of course, no alkalinity. An estimation of added tartaric acid, if any, is of some value, and may be carried out in the usual way, after first precipitating the organic acids as their lead salts in alcoholic solution, and decomposing these salts with sulphuretted hydrogen.

W. P. S.

Sitogen: a Vegetable "Meat Extract." **A. Beythien.** (*Zeit. für. Untersuch. der Nahr. und Genussmittel*, 1901, 446.)—In this paper is given an analysis of sitogen—an extract prepared from yeast. Coagulable albumen is practically absent. A quantitative analysis of the extract gave the following results:

Water	29·02 per cent.
Insoluble in water	0·38 „
Mineral matters	21·25 „
Phosphoric acid	5·56 „
Chlorine	5·19 „
Total nitrogen	7·01 „
Total acidity (as lactic acid)	3·38 „
Volatile acidity (as acetic acid)	0·16 „
Ether extract	0·74 „

The total nitrogen contained 0·43 per cent. present as ammonia (0·52 per cent.), 1·38 per cent. as albumoses (8·63 per cent.), and 5·15 per cent. as peptones and bases (32·19 per cent.). The mineral matter was composed of sodium chloride, 40·33 per cent.; phosphoric acid, 26·17 per cent.; sodium oxide, 15·12 per cent.; potassium oxide, 11·81 per cent., etc. The sodium chloride is added to the extract, and does not come from the yeast.

W. P. S.

Lloyd's Test for Morphine. **J. L. Mayer.** (*Amer. Journ. Pharm.*, 1901, lxxiii., 353-355.)—In this test a small amount of hydrastine is mixed with the substance and a few drops of strong sulphuric acid added, a violet-blue coloration being obtained after five minutes in the presence of morphine.

Conversely, hydrastine can be detected by the addition of morphine and sulphuric acid.

The author has applied this test to other alkaloids, using approximately 1 part of hydrastine with 8 parts of the given alkaloid, and has noted the final coloration after stirring the mixture for five minutes.

The following colorations were thus obtained: Aconitine, brown; atropine, pinkish; berberine, greenish-brown; brucine, light brown; caffeine, dirty white; cinchonine, dirty yellow; cinchonidine, dirty white; cocaine, unaffected; codeine, pinkish; digitaline, mahogany; heroine, violet to purple; homatropine, pale yellow; hyoscyamine, dirty white; morphine, violet-blue; pilocarpine, light brown; quinidine, light green; quinine, greenish-yellow; sparteine, greenish-yellow; strychnine, dirty white; veratrine, royal purple.

From this it appears that only heroine, morphine, and veratrine give a violet-blue colour in this test.

Of these, only veratrine gives a cherry-red colour with strong sulphuric acid; whilst morphine can be differentiated from heroine by means of nitric acid, the former giving an orange-red colour and the latter a yellow one. C. A. M.

Chemistry of the Jaborandi Alkaloids. **H. A. D. Jowett.** (*Brit. Med. Journ.*, October 13, 1900.)—The author has investigated the chemical properties of the four members of this class of alkaloids, the following being the results obtained:

Pilocarpine, $C_{11}H_{16}O_2N_2$, was formerly obtained from the leaves of true jaborandi (*Pilocarpus jaborandi*), but owing to the scarcity of these leaves the chief sources are the leaves of *P. pennatifolius* and Maranham jaborandi (*P. microphyllus*). These leaves may contain as much as 0·5 per cent. of the alkaloid. It is a thick syrup, yielding crystalline salts. The nitrate is fairly soluble in water, sparingly in strong alcohol. The hydrochloride is very soluble in water and moderately so in alcohol. The commercial nitrate is fairly pure, but contains a varying amount of isopilocarpine nitrate. The author suggests that, as standards of purity, the melting-point of the nitrate should be from 173° to 178°, and its specific rotation $+80°$ to $+83°$. As additional tests, the behaviour of a strong solution with excess of ammonia, and the formation of a crystalline picrate melting sharply at 147°, are suggested.

Isopilocarpine occurs with pilocarpine, and may be formed by heating the latter with water in a sealed tube for four hours at 180°. In its chemical properties it closely resembles pilocarpine, and the two cannot be easily separated by fractional precipitation. The greater amount of isopilocarpine found in pilocarpine is formed during the process of manufacture. In alkaline solution optically active pilocarpine is converted into the optically inactive isopilocarpine. In neutral solution isopilocarpine is optically active.

Pilocarpidine, $C_{10}H_{14}O_2N_2$, is found only in the leaves of true jaborandi. The nitrate is soluble in twice its own weight of water, and has a melting-point of 137°.

Jaborine.—No alkaloid answering to the description of jaborine is to be found in either true or Maranham jaborandi, and the jaborine of commerce is impure pilocarpine.

The Constitution of Pilocarpine.—Isopilocarpine, which, as is seen above, can readily be obtained by heating pilocarpine, acts not only as an ammonia base, but as a lactone, forms salts with hydrates, and can even be titrated with hot alkali. On oxidation it yields ammonia, methylamine, acetic acid, and an acid of the formula $C_7H_{10}O_4$, which is probably the lactone of hydroxy-isobutyl-malonic acid. Iso-butyric acid is formed by the fusion of pilocarpine with caustic potash. Methylamine alone is formed by the action of potash on methyl-isopilocarpine. There are thus indications of the following groups in isopilocarpine, $(CH_3)_2 : CH.CH.CH(CO_2)C : NH : NCH$ leaving three atoms of carbon and of hydrogen to be accounted for. W. P. S.

he Aniseed Oils and Anethol. G. F. Pancoast and L. F. Kebler. (*Amer. Pharm.*, 1901, lxxiii., 356-359.)—The seed from various sources yields from 6 per cent. of oil. Occasionally stems, chaff and leaves are added to the seed distillation. The chaff contains about 0·5 per cent. of oil.

is asserted that aniseed oil can only be distinguished from star anise (*Illicium*) by the odour and taste; and according to the authors none of the chemical re satisfactory.

tar anise oil is largely manufactured by the Chinese, and is exported in tin holding from 42 to 46 lb. Owing to its cheapness it has to a large extent ed the genuine oil.

he physical properties of star anise oil resemble those of aniseed oil, but its com- n is somewhat more complex.

he following results were obtained by the authors in the examination of various d oils sold as pure.

Source.	Specific Gravity.	Optical Rotation.	Congealing Point.	Solubility in Equal Volume of Absolute Alcohol.
ssian ...	0·9838 at 17° C.	+3° 50′	+15° C.	Soluble.
,, ...	0·9893 at 20° C.	−4° 59′	18° C.	,,
quin ...	0·9834 at 17° C.	−1° 30′	17° C.	,,
r anise ...	0·9648 ,,	−1° 27′	15° C.	,,
,, ...	0·9870 ,,	+0° 58′	16° C.	,,
,, ...	0·9822 ,,	−1° 53′	15·5° C.	,,
,, ...	0·9821 ,,	−1° 31′	14·5° C.	,,
,, ...	0·9832 ,,	−1° 44′	14° C.	,,

s regards these results, the authors considered that oils No. 1 and 2 had been red with, the first probably by the addition of oil of fennel. No. 8 was also mal, and possibly contained star anise leaf oil, which has a specific gravity of at 15° C., and an optical rotation of +1°. The amount of anethol in star leaf oil is small, and its congealing point correspondingly low.

ure oils with a low congealing point are sometimes met with. These are ed from a mixture of natural and artificially-ripened seed, and are known as ver Oils." In the author's opinion they are inferior to oil made entirely from eed.

nethol.—According to the German Pharmacopœia anethol is a colourless, refractive liquid, with an odour of pure anise. Its specific gravity at 25° C. 4 to 0·986. It melts at +20° to 21° C., boils at 232° to 234° C., and forms a solution with two parts of alcohol.

everal samples examined by the authors gave the following results:

Specific Gravity.	Optical Rotation.	Congealing Point.	Boiling Point.
0·9895 at 20° C.	inactive	+17° C.	210° to 235° C.
0·9896 at 20° C.	−1° 30′	+20° C.	220° to 235° C.
1·0525 at 15° C.	−2° 18′	—	228° to 245° C.
0·9870 at 20° C.	+5° 22′	+20° C.	229° to 236° C.
0·9847 at 20° C.	inactive	+19° C.	210° to 235° C.

All dissolved in an equal volume of alcohol. Of these, the authors regarded A, B, and E as of fair quality, although anethol is usually considered to be optically inactive. C and D were labelled "liquid anethol," by which is indicated a redistilled oil of anise prepared from ordinary aniseed oil. C was an old sample, and therefore had possibly altered in composition. D was considered to be "anethol" derived from oil of fennel.

C. A. M.

Saffron Adulteration and Saffron Essence. W. Fresenius and L. Grünhut. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1900, iii., 810.)—Two samples of adulterated saffron were examined by the authors with the following results:

	I.	Per Cent.
Adulterants	Magnesium sulphate ($MgSO_4$, $7H_2O$)	25·50
	Borax ($Na_2B_4O_7$, $10H_2O$)	8·23
	Neutral sodium borate ($Na_2B_2O_4$, $8H_2O$)	17·49
	Hygroscopic moisture	2·05
	Other constituents (difference)	46·73
		100·00

	II.	Per Cent.
Adulterants	Potassium nitrate	12·94
	Neutral potassium borate ($K_2B_2O_4$, $3H_2O$)	20·86
	Neutral sodium borate ($Na_2B_2O_4$, $4H_2O$)	6·41
	Sodium hydroxide	3·21
	Hygroscopic moisture	8·43
	Other constituents (difference)	48·15
		100·00

Caustic soda appears to have been added in both cases, in sufficient quantity in I. to convert part of the borax into neutral borate, and in II. to convert the whole into neutral borate and leave an excess, the object being apparently to increase the solubility of the added borax.

It is worthy of note that salts should have been selected which contain water of crystallization, which is only partially expelled on drying at 100° C.; thus, magnesium sulphate loses at 100° C. only 5 of its 7 molecules of water, and borax only 8 of its 10 molecules. A partial analysis, showing water lost at 100° C., ash and saffron by difference, would consequently be incorrect, and more particularly so since the determination of water lost at 100° C. would itself be inaccurate, owing to the absorption of carbonic acid by neutral sodium borate on drying.

The authors further examined a sample of so-called saffron essence, which has been placed upon the market. This article was found to have the following composition: Water, 46·57; borax crystals, 16·87; potassium hydroxide, 8·94; potassium nitrate, 10·03; saffron, 0·40; cane-sugar, 9·91; dextrose, 1·65; dextrin, 5·63. The authors suggest that it is simply the liquid which has been used for sophisticating saffron, as explained above, and that the saffron on being immersed in it has imparted to it some of its colouring and extractive matter.

H. H. B. S.

ORGANIC ANALYSIS.

Identification of "Renatured" Spirit. P. N. Raikow and P. Schtarbanow. (*Chem. Zeit.*, 1901, xxv., 434.)—This is a process for the detection and approximate estimation of methyl alcohol in renatured spirit, and depends (*a*) on the partial oxidation of the liquid to the respective aldehydes by means of a glowing platinum wire, (*b*) on the easy organoleptic detection of formaldehyde even in presence of a large excess of acetaldehyde by the mucous membranes of the nose and eye. The apparatus employed—it is important to observe the dimensions, etc., specified—consists of a conical flask of such size and charged with such a volume of liquid (say, a 100 c.c. flask and 10 to 20 c.c. of spirit) that the space not occupied by the alcohol shall be equal to 70 or 100 c.c., and shall be about 10 centimetres in height. In the neck of the flask is hung a spiral of platinum made of wire 0·3 millimetre in diameter. The alcohol is dehydrated by means of potassium hydroxide, and a small quantity is also put into the liquid to take up the water formed during the oxidation, but it is not necessary to remove the aqueous layer. The alcohol may be gently warmed, care being taken that its vapour do not expel all the air from the flask. The spiral is heated to redness in a flame, then dropped into position in the neck of the flask; it continues to glow steadily, and copious vapours are given off. The nose is held a few centimetres above the opening of the flask and the fumes are inhaled. From experiments on pure ethyl and methyl alcohol it appears that the vapours from the former can be breathed for minutes at a time; from methyl alcohol the vapour cannot be inspired twice. From 10 per cent. of methyl alcohol (90 per cent. of ethyl) inspiration is only possible for two or three seconds; from 2 per cent. thereof the peculiar odour of formaldehyde can be perceived in about fifteen seconds; the limit of delicacy by the nose being at about 1 per cent. The eye is somewhat more sensitive, but indications of 1 per cent. of methyl alcohol are doubtful. By distilling 100 c.c. of 99 volumes of ethyl mixed with 1 volume of methyl alcohol in an ordinary fractionating flask and collecting the first 10 c.c. which passes over, the odour is about equal to that of a liquid containing 2·5 per cent. of methyl alcohol. Similar experiments on German duty-free spirit from which the pyridine bases had been precipitated with phosphoric acid and the liquid concentrated (or not) by distillation were equally satisfactory.

The method may be rendered somewhat more elegant by placing the flask under an apparatus by the aid of which the evolved vapours can be aspirated through two flasks charged with water. The aldehydes condense, but the acetaldehyde can be easily driven off by heat or by continued aspiration, whereas the formaldehyde remains in the liquid, and will be found in the first condensing vessel. Fifty c.c. of pure ethyl alcohol may be oxidized in this apparatus (in nine hours), yielding liquids which reduce ammoniacal silver nitrate before or after evaporation to a small bulk, but which are free from the odour of formaldehyde. In presence of 1 per cent. of methyl alcohol the liquid in the first vessel will cause the eye to weep. It will be apparent that this test depends subjectively on the absolute quantity of formaldehyde in the liquid smelt, and that in the first form its chemical delicacy depends on the relative amount produced and volatilized in a given time, so that there is a distinct limit to its usefulness. In the second modification any quantity of alcohol can be decomposed by prolonging the time of treatment, and as the whole of the methyl alcohol will be recovered as formaldehyde, it serves to detect a smaller amount of the lower alcohol.

Preliminary experiments with ether and acetone show that in pure acetone the wire glows more brightly than in alcohol. The vapours have only a faint odour, which, like that of acetaldehyde, is felt at the anterior part of the nose, formaldehyde affecting the posterior portion. The vapours from acetone can be breathed for several minutes. One per cent. of methyl alcohol in acetone, accordingly, can be safely detected. With ether the spiral has to be supported just above the neck of the flask, when it will continue to glow for a few seconds. The vapours produced from ether are extremely irritating, but quite different from formaldehyde in odour, so that 2 per cent. of ether in acetone can be recognised. Two per cent. of ether in alcohol give a doubtful indication. The nature of the vapours evolved from acetone and ether is not yet known.
F. H. L.

Existence of Salicylic Acid in Wine. H. Mastbaum. (*Chem. Zeit.*, 1901, xxv., 465.)—In 1890 Medicus first called attention to the fact that certain undoubtedly genuine wines gave on extraction a substance yielding the violet colour with ferric chloride, which is supposed to be characteristic of salicylic acid. Sufficient of the material could not be obtained for the identification of its true nature; for, in fact, the test usually fails unless 100 or 200 c.c., or even a larger volume of wine is taken for examination. Hence, Medicus recommended that only 50 c.c. of a wine should be employed in seeking for added salicylic acid, and his proposal has been officially adopted in Germany. This method (extraction with equal volumes of ether and petroleum spirit) will indicate as little added salicylic acid as 0·5 gramme per hectolitre, *i.e.*, 0·25 milligramme per 50 c.c.

Mastbaum has attempted to discover whether Medicus' substance is actually salicylic acid, or whether, as Simon has suggested, it may be isopyrotritaric acid (*Compt. Rend.*, 1900, cxxxi., 618). A Torres Vedras wine was taken which was represented by the Tony-Garcin test to contain 0·9 milligramme of "salicylic acid" per litre. Five litres were concentrated to 500 c.c. and extracted with ether and petroleum spirit. Water was added, the volatile solvents removed, and the aqueous liquid treated with chloroform. From the latter a yellowish crystalline mass was obtained, which was dissolved in hot water, decolorized with animal charcoal, and filtered. The filtrate was extracted once with chloroform, afterwards with ether. These solutions, and especially the ether, yielded white needles which melted sharply at 155° C., sublimed above that temperature, and when they were dissolved in water gave the ferric chloride reaction strongly, also the Millon-Lintner test (ANALYST, 1900, xxv., 274), and Jorissen's reaction with sodium nitrite, etc. The yield was not large enough to determine the molecular weight and other constants, but there seems no doubt that the body is true salicylic acid, more particularly since some wines give the violet test more strongly after heating on the water-bath with 1 per cent. sulphuric acid than they do originally.
F. H. L.

A New Test for Sugar. T. Sollmann. (*Centralbl. Physiol.*, 1901, xv., 35; through *Chem. Zeit. Rep.*, 1901, 157.)—The reagent is a variant of Fehling's solution, prepared by mixing 50 c.c. of 10 per cent. sodium carbonate and 50 c.c. of 5 per cent. Rochelle salt with 10 c.c. of 1 per cent. cobalt nitrate or nickel sulphate. With nickel the reagent is apple-green; heated to boiling, and a little dextrose solution added, it gives a pale to canary-yellow colour, according to the proportion of sugar, the liquid remaining quite clear. The cobalt reagent is at first almost colourless, but in half an hour becomes bluish-green. Heated to the boil (alone), it turns sky-blue, becoming green again on cooling; soon a precipitate forms, and its delicacy falls off. If dextrose is added to the boiling cobalt reagent, the colour changes through emerald-green to yellow-green and reddish-brown. Traces of sugar produce only a pale green; large quantities yield a brown like iodine solution. These tints are permanent for days. The colours only develop at the boiling-point. The tests are one and a half to two times as delicate as those with copper.

The reactions succeed with dextrose, invert sugar, galactose, and the sugars produced by boiling gums with acids, also with aldehydes and various gums, the latter of which fail or give only a green colour with Fehling's solution. The reactions do not appear with saccharose, mannite, glycogen, dextrin, starch; aliphatic bodies such as acetone, alcohols, acids, and fats; aromatic substances, the xanthine group, creatinine, urea, alkaloids, glucosides, amido-acids, and inorganic reducing and oxidizing agents. With albuminoids colour changes appear, which, when typical, are different from the sugar tints; sometimes, however, they resemble them. F. H. L.

The Baudoin, Tambon, and Soltsien Reactions for Sesamé Oil. Utz. (*Chem. Zeit.*, 1901, xxv., 412.)—This article consists largely of a historical review of the Baudoin reaction, a copious list of references to the literature of that test being given. The author has examined Tambon's test (ANALYST, this vol., p. 105), using a 4 per cent. solution of dextrose in 1·19 hydrochloric acid. This reagent develops no colour of itself, at least in the cold, and it keeps well. On heating (alone) it gradually gives an orange colour, which, however, cannot be mistaken for the sesamé tint. With pure sesamé oil no colour appears on shaking in the cold for half a minute; on heating, the pink to red colour is produced, and attains its maximum in three to five minutes. The limit of delicacy appears to be at 2 per cent. of sesamé in an oil. Old or rancid sesamé gives a reaction: if pure, the colour is red-brown; mixed with other oils, pale brown. African sesamé yields the strongest colour, then Levantine oil, and finally Indian; this is the same phenomenon as obtains with the furfural test. Spectroscopically, the red Tambon mixture exhibits certain differences from the products of other tests; but the reaction cannot be recommended for general purposes.

Utz calls attention to the Soltsien test with stannous chloride. Properly carried out, it is the simplest, most certain, and most delicate of all; and it is particularly valuable when confirmatory evidence of the presence of sesamé is required.

F. H. L.

Apparatus for Determining the Melting-point of Gelatin-Jelly, Fats, etc. N. Chercheffsky. (*Chem. Zeit.*, 1901, xxv., 413.)—This apparatus consists of a 250 c.c. beaker filled with pale petroleum oil, and having a glass rod laid across its top. One end of a Z-shaped brass wire is coiled round the rod, the other depends into the liquid with its horizontal portion level with a thermometer bulb. On this end of the wire are skewered little cubes of the solidified gelatin solution, about 5 millimetres long, the jellies being prepared from equal weights of gelatin and water. For greate accuracy the beaker may be supported on cork blocks inside a water-jacket. B filling the inner vessel with water instead of oil, the melting-points of fats, waxes and the like may be similarly ascertained.

F. H. L.

A New Method for the Estimation of Soluble Nitrocellulose in Gun-cotto and Smokeless Powder. K. B. Quinan. (*Journ. Amer. Chem. Soc.*, xxiii., 258.)— The two methods now in use are the "aliquot" method, and the "residual" metho used by the United States Government. In the first, about 1 gramme of the drie and finely-divided sample is treated in a closed vessel with 250 c.c. of a mixture two parts of ether and one of alcohol. After all the soluble matter has been brough into solution by shaking, an aliquot part is transferred into a small tared flas evaporated at 65·5° C., dried at 100°, and weighed. This method gives good result but requires very careful working.

In the second method, 1·5 grammes of the sample are agitated in a covere vessel for at least two hours, with 250 c.c. of the same mixture of alcohol and ethe the residue is allowed to settle, the solution filtered through a tared asbestos filte the residue again treated with 200 c.c. of ether-alcohol, the solution filtered, and t asbestos filter and residue, after thorough washing, are dried first at 40° and then 100°, and weighed. The loss represents the soluble matter. By this method four five days are required for an analysis, rendering it practically useless for man facturers.

In the author's "centrifugal" method, 1 gramme of the sample is placed in small cylindrical aluminium vessel; 50 c.c. of alcohol are added, and the sample thoroughly stirred. After the addition of 100 c.c. of ether the whole is again stirre for several minutes, the vessel covered with a loosely-fitting cap, and placed in o of two cups mounted on the end of a horizontal shaft, which can be rotated at speed of about 2,000 revolutions per minute by means of a vertical driving-sha The power necessary for this is about ½ h.-p. In the other cup of the horizont arm a second sample may be placed, so that two analyses can be carried out at t same time. After the sample has been agitated for about ten or twelve minutes t aluminium vessel is removed from the cup, when it will be found that the insolub matter has settled completely at the bottom of the vessel. Almost the whole of t clear liquid can now be drawn off by means of a vacuum pipette provided with tw stopcocks in its lower and upper stems; the pipette is exhausted before use, and bo its stopcocks are closed; the lower stem is then immersed in the solution, and t lower stopcock opened, when the liquid will enter the pipette. To the residual 10 15 c.c. of solution in the aluminium vessel 50 to 75 c.c. of fresh ether-alcohol a added, the whole is stirred and again revolved in the centrifugal machine, and the liquid removed as before. This process is repeated until all the soluble matter has been removed, which can usually be done in seven or eight washings, though samples containing much insoluble matter may require twelve or more washings. The insoluble matter is then transferred to a Gooch crucible and dried at 100°, or dried directly in the aluminium vessel, and weighed.

Exclusive of the time required for drying, an analysis can be completed by this method in from one to two hours, as each successive extraction requires a shorter time in the centrifugal machine, since the viscosity of the solution decreases each time. The results obtained agree well with those given by the "aliquot" method.

A. G. L.

INORGANIC ANALYSIS.

The Measurement of Gold and Silver Buttons in Quantitative Blowpipe Assays. Joseph W. Richards. (*Journ. Amer. Chem. Soc.*, xxiii., 203.)—Beside actual inaccuracies in construction, the Plattner ivory scale possesses several defects, chief amongst which is the difficulty of getting the round buttons to the right spot on the flat surface of the scale. To remedy this, the author has contrived a scale consisting of two perfectly straight aluminium edges lying on a flat surface, touching each other at one end, and held apart at the other by a set-screw, so that the point 100 on the scale indicates a separation of 1 millimetre. In using the scale, the button is put into the groove, the scale inclined, and tapped until the button wedges itself in. Since the buttons are not perfectly spherical, the highest of several readings should be taken as the true horizontal diameter. In order to render them more uniform in shape, the cupelled buttons are remelted on charcoal before measuring, as recommended by V. Goldschmidt. One of the metal strips is pressed against the set-screw by a small spring which allows it to be pushed back, thus facilitating the removal of buttons and cleaning of the scale.

If the buttons consist only of gold or of silver, their weight may be obtained directly from a table given in the paper. If both gold and silver are present, the determination of silver is greatly simplified by the use of the case of standard alloys designed by Goldschmidt. In the first place the button is measured, and its volume obtained from the table. It is then flattened out and compared with the standard alloys, under a lens, by diffused daylight. The percentage of silver is thus determined approximately. The specific gravity of the alloy is then obtained from a table also given in the paper, and, on multiplying the volume by this, the weight of the original button is obtained, from which the weight of gold and silver can be found.

This method cannot be applied to buttons containing more than 56 per cent. of silver, since all these alloys are silver-white. In this case the best plan is to measure the button and obtain its volume from the table, then replace it on charcoal, and heat intensely in the oxidizing flame, when the silver will slowly volatilize. As soon as the button has a pronounced yellow colour, which will be the case after one to five minutes, it is again measured, flattened out, and compared with the standard alloys. The weight of gold present may then be found as before, and on taking the volume corresponding to this weight from the volume of original button, the volume of silver present is obtained, from which its weight may be calculated. Another method consists in melting the original button with a button of pure gold of about the same (known) diameter. The button obtained is measured, compared with the standard buttons, and the weights of silver and gold obtained as before. The weight of gold added is then subtracted from the total weight of gold, and the remainder gives the weight of gold in the assay. All these methods are based on the fact, which has been experimentally verified, that gold and silver neither expand nor contract on alloying with each other. A. G. L.

Estimation of Zinc with Iodine Solution. P. Knaps. (*Chem. Zeit.*, 1901, xxv., 539.)—In acetic acid solution, zinc sulphide reacts quantitatively with iodine to form zinc iodide and sulphur. But in solutions containing more than 0·05 gramme per 200 c.c. of water, part of the sulphide and the liberated sulphur cohere, and prevent the completion of the decomposition. This, however, can be avoided by producing within the liquid some indifferent precipitate such as barium sulphate, which iodine does not attack. The original zinc solution is treated with 10 c.c. (for every 0·05 gramme of zinc) of a solution of 150 grammes of crystallized barium chloride per litre, and an equal volume of a solution of 200 grammes of crystalline sodium sulphate per litre is added. The zinc is then thrown down with sulphuretted hydrogen, the excess of the gas is driven off by sharp boiling till a lead paper is no longer discoloured by the vapours, the liquid is cooled, excess of standard iodine is run in, shaken for a minute or two, and the whole titrated, as it is, with thiosulphate. Reasonable amounts of manganese salts do not interfere; nitrates must be absent. The results quoted appear satisfactory, even in presence of manganese. F. H. L.

Detection of Indium. P. Kley. (*Chem. Zeit.*, 1901, xxv., 563.)—The best substance for the detection of indium is the double chloride of that metal with rubidium, which is produced by adding a crystal of rubidium chloride to a solution of indium hydroxide in strong hydrochloric acid. The crystals appear as colourless octahedra which polarize light and belong to the rhombic system; they measure 10 to 70 μ. The limit of delicacy of the reaction is at 0·00024 milligramme of indium. By employing cæsium chloride instead of rubidium chloride, the test is ten times more delicate, but the crystals are so small as to be difficult of recognition. Indium can be separated from other metals by treatment with ammonia; the hydroxide adheres to the microscope object glass, and can be washed with ammonia and water till the rest of the zinc, cadmium, etc., is removed. Neither aluminium nor zinc affects the process. If 50 per cent. of zinc is mixed with the indium, the precipitation with ammonia is not necessary; but if the indium is accompanied by large amounts of trivalent metals, the liquid should be treated with ammonia, the indium hydroxide dissolved in ammonium carbonate, and then thrown down with oxalic acid. When, however, the quantity of indium is extremely minute, the rubidium chloride reaction is better; but all ammonia, and as much ammonium chloride as possible, should be removed by heating, or the crystals will be very small.

n chloride yields a double salt with indium chloride as rubidium and . The ammonium compound is readily soluble in hydrochloric acid, like ium compound.

se has recently recommended a micro-chemical test for indium based on the of cæsium-indium alum. Even in strong solutions indium sulphate with lphuric acid yields the above-mentioned double chloride when cæsium added. The reaction succeeds under similar conditions if cæsium nitrate ; but it is useless in presence of aluminium. Kley has not been able to reaction with ammonium mercuric thiocyanate, also mentioned by Huysse, ghly concentrated solutions. Hitherto this reaction has only been known ith divalent metals. Huysse's description reads like that of the corre-inc salt; and as indium is recovered from zinc-dust, it seems possible that cause of the reaction. Huysse's oxalate test only works in ammoniacal nd is valueless when but little indium is present. F. H. L.

---

metric Estimation of Chromium in Chrome Mordants. R. Hartmann. *it.*, 1901, xxv., 564.)—About 1 gramme of the chrome acetate liquor (of 21° s diluted with 10 or 15 c.c. of water, and in a 1-litre porcelain basin is th strong sodium hydroxide till the precipitate redissolves. At the tempera-water-bath sodium peroxide is next added until the liquid is pure yellow. evaporated to dryness in order to destroy traces of peroxides, dissolved in water, made acid with dilute sulphuric acid, and mixed with 50 c.c. of l ferrous ammonium sulphate. Finally the solution is diluted to 1 litre, d with permanganate till the green colour changes to a dirty violet. At on prescribed, the end-point is quite sharp. Acetate prepared from e and molasses, starch, etc., must first be freed from reducing matter by itric acid or hydrochloric acid and chlorate, then converted into sulphate The organic matter in commercial acetate reacts with permanganate, and amount of chromium oxide to appear a little too low, but the error is to 0·09 per cent., and for commercial work may be ignored. The results satisfactory. F. H. L.

---

he Quantitative Precipitation of Metals by Organic Bases. W. Herz. *rg. Chem.*, xxvii., 310.)—In pursuing his experiments on the precipitation of organic bases possessing high dissociation constants (*Zeit. anorg. Chem.*, and 347), the author found that copper could be quantitatively precipitated a solution of guanidine or piperidine to the solution of the copper salt, arming for a short time, filtering the precipitate, and igniting as usual to gnesium and zinc were also completely precipitated from solutions of their by the addition of a solution of piperidine, and allowing to stand for some re filtering, but a little $Zn(OH)_2$ is apt to go through the filter, leading to s. In each case the result of only one experiment is given.

azine cannot be employed, as its dissociation constant is too small (0·0064). ernary ammonium bases, *e.g.*, tetramethyl-ammonium hydroxide, which possess high dissociation constants (0·211), also precipitate magnesium, copper, and zinc salts completely, but the precipitates are so gelatinous that it is almost impossible to wash them completely, and consequently the results obtained are too high.

A. G. L.

Analysis of Cement. O. H. Klein and S. F. Peckman. (*Journ. Soc. Chem. Ind.*, 1901, 539.)—The sample for analysis should not be pulverized, but should be exactly like the sample submitted to physical tests. Five grammes are weighed out and gradually introduced into 250 c.c. of not stronger than 10 per cent. hydrochloric acid in such a manner as to avoid any appreciable rise in temperature. The solution should be vigorously stirred at intervals for half an hour. The residue consists of unburnt fuel and ashes. It is separated and weighed. The filtered solution is evaporated to dryness, drenched with concentrated hydrochloric acid, taken up in water, the "soluble silica" filtered off, dried and ignited. The filtrate is made up to 1 litre, and two portions of 100 c.c. each are precipitated with ammonia. The precipitate is filtered off, redissolved in hydrochloric acid, and reprecipitated with ammonia. In the filtrate lime is precipitated as oxalate. Magnesia is next separated. The sulphur may be determined either in the filtrate from the magnesia, or better in the original solution. The only other determination the authors make is the loss on ignition. The "soluble silica," the alumina and iron oxide and the lime, which together constitute the "true cement," should amount to about 90 per cent. The "insoluble in 10 per cent. HCl" in good cements amounts to 3 to 5 per cent., and is considered by the authors to be very harmful. The other impurities they consider to be comparatively harmless.

A. M.

Estimation of Citrate-soluble Phosphoric Acid in Thomas Meal by the Molybdate Method. O. Foerster. (*Chem. Zeit.*, 1901, xxv., 421.)—According to Wagner's directions, the citric acid solution of the phosphoric acid must be placed for ten to fifteen minutes in a water-bath heated to between 80° and 95° C. after adding the ammonium molybdate; and the yellow precipitate must be quickly and entirely soluble in 2 per cent. ammonia. These two regulations are somewhat antagonistic; if the bath is maintained at the temperature quoted, complete solubility is but seldom attained. It is better to put the beaker in a water-bath which has been heated to 80° C., allowing the temperature to fall naturally during the ten or fifteen minutes' immersion, for in that way precipitation of silica is reduced to an insignificant amount. Even a temperature of 60° C. is sufficient to throw down all the phosphoric acid.

The quantity of silica precipitated from the same sample at the same temperature is not always the same, but varies for reasons which have not yet been explained. A thorough investigation of the effect of temperature on the molybdate process would be very welcome.

F. H. L.

ERRATA.

Page 229, 5th line from bottom, *for* "As the figures . . . ." *read* "As the figures given for a commercial malt extract must necessarily be of a purely empirical nature, it was decided . . . ."

Page 230, foot-note, *for* "author's" *read* "authors'."

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THE ANALYST.

NOVEMBER, 1901.

THE INCUBATOR TEST FOR SEWAGE EFFLUENTS.

By F. Wallis Stoddart, F.I.C.

The incubator test stands alone amongst the various means devised for determining the quality of a sewage effluent, in that it affords a direct and conclusive proof of conformity, or the reverse, with the requirements of the Rivers Pollution and Public Health Acts. Offences against these Acts, with the exception of the improper deposition of solids, are the result of putrefactive changes. The ordinary analysis of an effluent and its relation to dissolved oxygen afford valuable but only indirect evidence of its liability to putrefaction, the deductions drawn from the analytical data being further based upon premises not universally accepted, whilst the analysis itself is far too cumbrous a performance to apply sufficiently often to give a reliable insight into the character of so variable a liquid as sewage effluents commonly are; the incubator test, on the other hand, goes at once to the root of the matter, can be applied to any reasonable number of samples, and gives results which require no further elaboration.

The method of carrying out this test is thus described in the "Experts' Report on Treatment of Manchester Sewage": "A determination is first made of the oxygen absorbed from potassium permanganate by the sample in three minutes. A bottle is then completely filled with the sample, and placed in the incubator at 80° F. for six or seven days. The three minutes' oxygen absorption is then again determined. If any putrefaction has taken place, the oxygen absorbed in three minutes will exhibit a decided increase in amount owing to the more ready oxidizability of the products of putrefaction, such as sulphuretted hydrogen, etc. On the other hand, if the sample keeps sweet, the three minutes' oxygen absorption remains practically unchanged after incubation, or there will be a slight decrease owing to slight oxidation of the impurities which has taken place during the period of incubation at the expense of the nitrate or dissolved air present in the sample.

The test under these conditions is not entirely satisfactory, and does not distinguish with sufficient sharpness between good and bad effluents, as may be seen on reference to the Manchester report, in which the increase in oxygen absorption by unfiltered tank effluent rarely reaches 50 per cent., and even the crude sewage occasionally shows complete stability.

With the view of improving this test, and reducing, if possible, the length of incubation, I have carried out a number of experiments with sewage produc various kinds, and, chiefly by raising the temperature of incubation, have succe in so far emphasizing the distinction between putrescible and imputrescible liqui to have arrived at a test which might well be adopted in connection with any fu legislative measures. The liquid to be tested should be enclosed in well-stopp bottles, which should be quite full. Mercury valves are objectionable, as the mer is attacked and fixes an appreciable amount of the sulphides formed in putrefac The incubating temperature should be 37° C., when much more active changes ensue than at 27° C., resulting in a much more copious production of redu bodies. The bottle should be well shaken before the measured quantity is take the estimation of absorbed oxygen.

With unfiltered tank effluent a marked increase in absorption is noted after t days, but the change is much more pronounced at the end of a week, and conti for a considerably longer period.

It is found that the formation of sulphide runs parallel with, and is no doubt chief cause of, the increase in oxygen absorption; this being so, the test may be further simplified by substituting the addition of lead solution for the permanga process, and, if necessary, a colorimetric estimation may be made, for which pur a solution of lead chloride will be found to be preferable to the usual acetate. all practical purposes, however, a visible darkening on addition of the lead test incubation for from three to six days is all that is required, and this is the for criterion I should suggest for official purposes; any effluent that will give eviden putrescibility under the current test will be readily detected in this way. Indeed modified test is, if anything, too severe, and will be found to condemn many efflu which comply with the usual standards; as, however, it is now possible by mo methods to continuously convert sewage into a liquid capable of passing the mod test at a single expenditure of a few shillings per head of population, no injustice would be inflicted by its adoption.

The following table illustrates the application of this test to unfiltered preci tion and septic effluents:

INCUBATOR TEST.

Three Minutes' Oxygen Absorption of Unfiltered Precipitation and Septic Effluen before and after Incubation at 37° C. in full Bottles.

Length of Incubation in Days.	Fresh.	1.	2.	3.	4.	5.	7.	8.	9.	10.	11.
Precipitation effluent ...	0·700	0·700	0·727	0·952	1·30	1·40	1·456	1·624	1·638	1·68	1·736
Percentage increase ...	—	—	12	36	86	100	108	132	134	140	148
Septic effluent	0·560	0·560	0·694	1·12	1·68	1·89	1·994	2·036	2·13	2·156	2·268
Percentage increase ..	—	—	24	100	200	236	256	264	280	285	305

The formation of sulphide as indicated by the lead test is exactly parallel with the oxygen absorption.

It would be anticipated on theoretical grounds that the septic product would be a more putrescible and offensive liquid than the precipitation effluent, and that if treatment were limited to tank operations, as is even now sometimes suggested, chemical purification would be more effective than septic action alone. It is interesting to note the more copious and rapid production of oxidizable (and offensive) bodies in the much weaker septic effluent in the above table, a change which, however, presents marked advantages where the tank product is applied to the aerating filter now universally recommended.

COMPOUND TINCTURE OF BENZOIN.

BY ALFRED HILL, M.D., F.I.C., AND J. F. LIVERSEEGE, F.I.C., PH.C.

THE British Pharmacopœia of 1898 orders that compound tincture of benzoin shall be prepared from benzoin, prepared storax, balsam of tolu, Socotrine aloes, and 90 per cent. alcohol.

Benzoin.—The requirement of the Pharmacopœia is that benzoin shall be "almost entirely soluble in alcohol (90 per cent.)." The best quality of benzoin is Siam, and, according to Dieterich (*Chemist and Druggist*, November, 1898), from 95 to 100 per cent. is soluble in alcohol. Sumatra benzoin contains from 7·1 to 31·9 per cent. of insoluble matter (Evans, *Pharmaceutical Journal*, May, 1898; Dieterich, *Helfenberger Annalen*). The editor of the Pharmacopœia, in his 1898 Report, remarks:

"For official use practically no such insoluble matter is permissible; benzoin must be 'almost entirely soluble.' Until it has been shown that the bark, etc., has parted with harmful soluble matter to the alcohol used in preparing the official compound tincture, benzoin containing the usual varying proportions of bark (1 to 30 per cent.) may be employed, but allowance must, of course, be made for the insoluble matter, so that 1 pint of the official tincture shall be prepared from 2 ounces of *benzoin*, and not from 2 ounces of benzoin and bark."

Twelve samples of Sumatra benzoin examined by Dieterich lost from 4·6 to 8·2 per cent. by drying at 100° C. A single sample examined by us, after solution in alcohol, lost 4·6 per cent. of its weight on drying in the water-oven.

From the above results it seems reasonable to expect that in the preparation of the tincture 100 parts of official benzoin should yield soluble matter equivalent to at least 85 parts of dry extract, allowing a loss of 15 per cent. of insoluble and volatile matter.

Storax.—Crude storax contains from 1·4 to 7·3 per cent. of matter insoluble in alcohol, and from 19·6 to 41 per cent. of water (Dieterich, *Pharm. Central.*, 40, 427). The Pharmacopœia preparation is obtained from this "by solution in ethylic alcohol, filtration, and evaporation of the solvent." It is officially required to give off no moisture on heating in a test-tube. Eight samples of prepared storax examined Dieterich lost from 5·1 to 12·6 per cent. by drying at 100° C. He considers that t moisture should not exceed 8 per cent. (*Chemist and Druggist*, July, 1898). T United States Pharmacopœia requires that storax, after solution in alcohol a evaporation of the solvent, shall yield at least 70 per cent. of solid residue. As t British Pharmacopœia article has been purified, probably 80 per cent. of it will appe as dry extract when used for the tincture.

Balsam of Tolu.—The British Pharmacopœia requires that it shall be soluble alcohol. Five samples examined by Dieterich conformed with this requirement. sample examined by us after solution in alcohol lost 6·2 per cent. of its weight o drying at 100° C. Probably an allowance of 10 per cent. for volatile matter woul be sufficient.

Socotrine Aloes.—The Pharmacopœia requires this drug to be almost entirel soluble in 45 per cent. alcohol. Hager (ANALYST, x. 95) states that it is completel soluble in 80 per cent. alcohol, but only partially in 90 per cent. When a gramme o Socotrine aloes was treated with 50 c.c. of 90 per cent. alcohol, as in making th tincture, and filtered, we found that the filtrate, after evaporation of the solvent an drying in the water-oven for three hours, yielded 97 per cent. of the weight taken i the form of dry extract. Probably, therefore, an allowance of 10 per cent. fo insoluble and volatile matter is ample.

Quantity of Extract in the Tincture.—In the following table the quantities of th ingredients ordered by the Pharmacopœia for 1 litre of the tincture are given, and b the use of the standards given above the amount of the solid extract which shoul be yielded by each is calculated :

Quantities given by the Pharmacopœia.		Amount of Extract in	
		Drug.	Tincture.
	Grammes.	Per Cent.	Grammes.
Benzoin ...	... 100	85	85
Prepared storax ...	75	80	60
Balsam of tolu ...	25	90	22·5
Socotrine aloes ...	18·3	90	16·5
Per litre ...	... 218·3		184

We think that the above statement justifies the adoption of a standard 180 grammes per litre as a reasonable one. It must be remembered that deficie in one drug may be compensated by the good quality of the others. A tinct prepared by us without any special attention being given to the quality of the dr contained 182 grammes of solid extract per litre. The extract was determined evaporating 5 c.c. of the tincture to dryness on the water-bath in a German-sil dish 3 inches in diameter, with a flat bottom, and drying the residue three hours the water-oven.

Twenty-five samples of compound tincture of benzoin purchased officially by t

Birmingham Food and Drugs Inspector during the last two years yielded the following amounts of solid extract:

Grammes Solid Extract Per Litre.	Number of Samples.	
201	1	
180 to 187	13	
174 to 178	7	
169	1	
158	1	
147	1	(Vendor was fined £5)
115	1	(Vendor was fined £20)

The average of the whole of the samples is 176 grammes of solid extract per litre, or, if the two obviously adulterated ones be omitted, 180 grammes. The sample with 158 grammes was passed as of low quality. Probably 160 grammes may be taken as the minimum limit for tincture of benzoin.

The standard of 180 grammes per litre is advocated by Moor and Priest (*Chemist and Druggist*, July, 1900). A sample prepared by Seyler (Report to the Glamorgan County Council, 1897) yielded 175 grammes. Merson, in a paper read this year before the Pharmaceutical Society, states that as a result of eight experiments, using all grades both of benzoin and storax, he considered not less than 170 to 180 grammes of solids per litre should be present. His mean result was 178 grammes. He points out that if storax is used, instead of the official *prepared storax*, less than 160 grammes may be present even if good Siam benzoin is used, and that a tincture prepared with a moderate Sumatra benzoin and prepared storax gave 181·5 grammes of solid matter per litre (*Pharmaceutical Journal*, February 23). At the recent meeting of the British Pharmaceutical Conference, H. Whippell Gadd stated that if the extractive falls much below 180 grammes per litre, it is evident that an inferior benzoin or storax, or both, have been used. F. W. Fletcher (*Chemist and Druggist*, January, 1901) gives 15·8 grammes as the "percentage of solid residue." If by "percentage" he means grammes per 100 c.c. the result is very low for a standard tincture, but if it is a true percentage, as the specific gravity of the tincture is given as 0·892, it will indicate 177 grammes per litre.

Specific Gravity of the Tincture.—The specific gravity of the tincture prepared by us was 0·896 at 60° C. The specific gravities of the twenty-five official samples were as follows:

Specific Gravity at 60° F.	Number of Samples.	
0·906-0·904	3	
0·903-0·900	8	
0·899-0·895	9	
0·893	2	
0·891	1	
0·886	1	(Vendor was fined £5)
0·880	1	(Vendor was fined £20)

In the next table are given various published specific gravities of the 1898 compound tincture of benzoin.

Specific Gravity.	Reference.
0·893	J. C. Umney. *Chemist and Druggist*, April, 1898.
0·894	J. Attfield. Report on Pharmacopœia for 1898.
0·900	J. Barclay. *Chemist and Druggist*, December, 1898.
0·902-0·907	(" Allowance made for insoluble matter in the benzoin to accord with the British Pharmacopœia monograph, otherwise tincture is lighter.") Holthouse and Harvey. *Chemist and Druggist*, July, 1900.
0·890-0·900	Moor and Priest. *Chemist and Druggist*, July, 1901.
0·890-0·900	Year-Book of Pharmacy, 1900.
0·892	F. W. Fletcher. *Chemist and Druggist*, January, 1901.
0·8983	Merson. *Pharmaceutical Journal*, February, 1901.

The foregoing results are of considerable importance, in view of a case which has come before the magistrates at Birmingham. A sample of Pharmacopœial tincture of benzoin purchased under the Sale of Food and Drugs Act showed in two determinations a specific gravity of 0·8840 and 0·8846, and 146 and 147 grammes per litre. A certificate was given by the Public Analyst to the effect that the sample contained only 82 per cent. of the quantity of solid matter ordinarily present in tincture of benzoin. The inspector's sample having been referred to the chemists of the Government Laboratory, a report was received from them stating that " in our opinion it affords no evidence of being below the strength of the compound tincture of benzoin made according to the British Pharmacopœia of 1898." By the courtesy of the Chief of the Government Laboratory, we are enabled to state that the sample in question was found by the Government chemists to contain 133 per cent. of proof spirit by volume, and yielded 16·4 per cent. of extract dried at 100° C. Calculation shows that 147 grammes per litre is equal to 16·6 per cent. by weight, which is in practical agreement with the quantity found at the Government Laboratory. Reference to the figures given in the earlier portion of the paper shows that the quantity should be considerably higher than this.

THE RECIPROCAL DETERMINATION OF CYANIDES AND CYANATES.

By J. W. Mellor, B.Sc. N.Z. University.

Enormous quantities of potassium cyanide are used in certain gold-extraction processes. It is well known that the commercial article contains more or less cyanate, which appears to be useless from a metallurgical point of view. In the course of some experiments on solutions of potassium cyanide, it was desired to estimate the quantity of potassium cyanate in the presence of cyanide and *vice versâ*.

F. Wöhler* has pointed out that silver cyanate is decomposed by dilute nitric acid thus:

$$AgOCN + 2HNO_3 + H_2O = AgNO_3 + NH_4NO_3 + CO_2.$$

The addition of nitric acid before the usual titration, however, interferes with the accuracy of the test, since the double cyanide of silver and potassium is decomposed by the dilute acid, with evolution of hydrogen cyanide and the precipitation of silver cyanide.

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* Wöhler: Gilbert's *Ann. d. Phys.*, 73, 157, 1823.

A. H. Allen[*] has employed Wöhler's reaction for the determination of cyanates Since 1 molecule of silver cyanate neutralizes 2 molecules of nitric acid, if excess of normal acid be added to the cyanate, on titrating back with normal alkali, 1 c.c. normal HNO_3 neutralized by the cyanate represents 0·0405 gramme potassium cyanate.

G. Denigés[†] has improved the ordinary cyanide determination by Liebig's process, by titrating an ammoniacal cyanide solution with $\frac{N}{10}$ $AgNO_3$, using 10 drops of a 20 per cent. aqueous solution of KI as indicator. The sparing solubility of AgI in aqueous ammonia makes the end-reaction much sharper. As usual, one equivalent of $AgNO_3$ represents two equivalents of KCN.

Since silver cyanate and cyanide are both much more soluble in aqueous ammonia than silver iodide, Denigés' process may be applied. The presence of KOCN appears to make no difference to the cyanide determination under these conditions—*e.g.* :

> Ten c.c. KCN solution required 14·3 c.c. $\frac{N}{10}$ $AgNO_3$; 10 c.c. of the same solution, with 10 c.c. of KOCN solution added, required 14·3 c.c. $\frac{N}{10}$ $AgNO_3$; and similarly with 40 c.c. of the same solution of KOCN added, 14·4 c.c. $\frac{N}{10}$ $AgNO_3$ was necessary.

Thus, Denigés' and Allen's processes are reciprocal for the determination of cyanates and cyanides in the presence of one another.

The principle of the proposed method for the determination of cyanides and of cyanates is first to determine the equivalent of the cyanide in terms of silver nitrate, and then to estimate the amount of cyanate in the whole in terms of nitric acid.

The following *modus operandi* was adopted with good results :

1. Dissolve 20 grammes commercial potassium cyanide in about 100 c.c. of distilled water. Add a solution of calcium nitrate—free from chlorides—to precipitate any carbonates that may be present. Barium nitrate cannot be used on account of the insolubility of barium cyanate. Filter and wash the precipitate with distilled water. Collect the filtrate and washings in a 200 c.c. flask, and make up to 200 c.c. One c.c. of this solution represents 0·1 gramme of the original sample.

The precipitate contains insoluble impurities (carbonaceous and earthy matters), carbonate and sulphate of calcium, etc. If desired, each of these may be determined by the usual methods of analysis.

2. Mix 10 c.c. of the above solution of the sample of cyanide with excess of aqueous ammonia and a few drops of KI solution. Titrate with $\frac{N}{10}$ $AgNO_3$ solution, and suppose that *m* c.c. of this solution are required.

3. Run concentrated $AgNO_3$ solution into another portion—say, 10 c.c. of the above aqueous solution of cyanide—until no further precipitation occurs. Thus, all the cyanide radicle in the $KAgCy_2$ is precipitated as AgCN. Filter and wash the precipitate of mixed cyanide and cyanate of silver with ice-cold distilled water. Add,

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[*] Allen's "Commercial Organic Analysis," 1896, 3, iii., 484. This observation appears to be the only published record on the determination of cyanates in the presence of cyanides.

[†] Denigés, *Ann. de Chim. et de Phys.* [7], 6, 381, 1895. An abstract occurs in the *Journ. Chem. Soc.*, 1896.

say, 5 c.c. normal HNO_3 and warm to about 50°, when any cyanate will be decomposed. Filter and wash. Treat the filtrate with standard alkali. Suppose that n c.c. of normal NaOH solution are required, then 0·0405 $(5-n)$ gramme (or 4·05 $[5-n]$ per cent.) KOCN are contained in 1 gramme of sample; 0·013 m gramme (or 1·3 m gramme per cent.) KCN are contained in 1 gramme of sample.

If desired, the results can be expressed as "available cyanogen"; from 0·0052 m gramme (or 0·52 m gramme per cent.) available cyanogen are contained in 1 gramme of sample.

Instead of using 10 c.c. of the original solution in 3 above, q c.c. may be used when there is 0·00405 $(5-n)$ q gramme KOCN in 1 gramme of sample.

If n' c.c. of $\frac{N}{10}$ alkali be used, then 10 n' must be substituted for n unless $\frac{N}{10}$ acid has also been employed.

If p c.c. of nitric acid be used instead of 5 c.c. in the decomposition of silver cyanate, then there is 0·0405 $(p-n)$ gramme KOCN in 1 gramme of sample.

The presence of sodium cyanide introduces a considerable error in the calculation of available cyanogen to potassium cyanide alone. The maximum amount of available cyanogen in pure KCN is 40 per cent., while for NaCN the maximum is 53 per cent.; hence a little of the latter present would calculate to more KCN than was actually present. A sample containing 15 per cent. NaCN would be reported as 100 per cent. KCN.* This would be an unsatisfactory valuation unless sodium cyanide is at least equal to the potassium salt in its power of extracting gold from "tailings."

Chlorides, if present, may be estimated as follows: Fuse in a porcelain basin a known weight of sample, with a mixture of KNO_3 and Na_2CO_3 (1 : 5) carefully added. Boil the fused mass with water acidulated with HNO_3, and estimate the chlorides in solution in the usual way.†

The following is an analysis made during October, 1897, in the Otago University College Laboratory, New Zealand, of a specimen obtained from a gold extraction company such as was in use in their cyanide works. No sulphides, thiocyanates, or formates were present:‡

		Percentage Amounts.
Insoluble matter	Carbonaceous	4·35
	Ash	1·07
Carbonate radicle (CO_3)		4·69
Sulphate radicle (SO_4)		traces
Chloride radicle (Cl)		traces
Cyanate radicle (OCN)		4·04
Cyanide radicle (CN)		32·51
Potassium		49·90
Sodium		1·59
Loss		1·85

The loss is probably due to moisture, although the specimen stood over $CaCl_2$ for twenty-four hours before weighing. It seems impossible to determine to which negative radicles Na or K must be calculated; hence the advantage of recording results as "available cyanogen."

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* Kayser, *Chem. Zeitung*, 16, 1148, 1892.

† Watts' "Dictionary of Chemistry," 1889, 846.

‡ *Ibid.*; *cf.* also Denigès, *loc. cit.*

With artificially-prepared mixtures of "pure" potassium cyanide by Wiggers' process,[*] and of potassium cyanate by Bell's process,[†] an average error of +0·18±0·01 per cent. in the determination of KOCN was obtained when the components of the mixture were assumed to be absolutely pure. The constancy of the error, however, would make this appear doubtful. More recent determinations with KOCN and KCN of known purity gave an average error of 0·04±0·01 per cent. in ten experiments.

SYSTEMATIC INSPECTION OF MILK FOR PRESERVATIVES.

By Albert E. Leach, S.B.

(*Analyst of the Massachusetts State Board of Health*).

In the June number of the Analyst for the current year (p. 148) appeared a paper by M. Wynter Blyth on "The Detection and Estimation of Preservatives in Milk," in which was described a method for subjecting all the samples to certain fixed bacterial conditions during a period of some twenty-four hours, with a view to selecting those samples which contained preservatives, and afterwards identifying the particular preservative present.

It occurred to the writer that it might not be without interest in this connection to English analysts to know how, as a matter of routine, a large number of milk samples are systematically subjected to tests for preservatives daily throughout the summer in the laboratory of the Department of Food and Drug Inspection of the Massachusetts State Board of Health.

All the milk samples collected by three inspectors throughout the State, with the exception of four western counties, are analysed in the laboratory of the Department at Boston. These amount to upwards of 500 samples per month, the daily number analysed varying from 10 to 60, and during the months of June, July, August, and September, in addition to the regular analysis for total solids and fat, all are systematically examined for preservatives by methods which during four years have proved both quick and accurate.

At the outset it should be said that the only preservatives found in milk in Massachusetts at the present time are formaldehyde, boracic acid and borax, and sodium bicarbonate. Salicylic acid has not been found in milk in this State for many years. It is a very poor milk preservative, on account of the taste imparted to the milk, and it is difficult to conceive why it should be used at all at the present time for this purpose, in view of the much more efficient antiseptics at hand.

The following summary shows the extent of the use of antiseptic substances in milk in Massachusetts during the last three years:

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[*] Wiggers, *Liebig Ann.*, 1839, 29, 65.

[†] Bell, *Chem. News*, 1875, 32, 99.

Year.	Samples Examined.	No. containing Formaldehyde.	Per Cent. containing Formaldehyde.	No. containing Boracic Acid.	Per Cent. containing Boracic Acid.	No. containing Carbonate.	Per Cent. containing Carbonate.	Total co taining Preserv tive.
1898* ...	1,046	26	2·5	11	1·0	4	0·4	41
1899† ...	2,105	55	2·6	13	0·6	3	0·3	71
1900† ...	2,018	61	3·0	6	0·3	—	—	67
Total	5,169	142	2·7	30	0·6	7	0·1	179

Thus, out of 5,169 samples examined for preservatives, 179, or 3·5 per cen were found to contain them. It will be seen from the above table that the use boracic acid and sodium bicarbonate is on the decrease, while formaldehyde is bein used more and more. This is to be expected, in view of the fact that formaldehyd being in solution, lends itself more naturally to the milkman's immediate use, beside being the most active of preservatives, and imparting no appreciable taste to th milk, even when used in considerable strength.

The method of procedure is as follows: After the milk samples have bee analysed for total solids and fat, they are arranged in order, and about 10 c.c. o each sample is poured into a porcelain basin, the basins being, for convenience arranged in the same order as the containers of the milk samples. Each casserole i then treated alike in succession, first adding, from a large stock-bottle provided wit a delivery siphon and stopcock, about 10 c.c. of the hydrochloric reagent, and the heating directly over the gas-flame, holding the basin by the handle, and giving i a rotary motion during the heating to break up the curd and hasten its solution i the acid. The reagent contained in the stock-bottle consists of commercial hydrochloric acid (specific gravity, 1·20) containing 1 c.c. of 10 per cent. ferric chlorid solution to each 500 c.c. of acid. The heating is continued nearly to the boiling point. The presence of formaldehyde is indicated by the well-known violet coloration, varying in depth with the amount present. By this test it has been found th 1 part of formaldehyde in 500,000 parts of milk is readily detected before the mi has soured. After souring, the limit of delicacy proves to be about 1 part in 50,00 In point of actual time consumed in making the test, less than one minute sufficient for each sample.

The hydrochloric acid method has been the subject of much criticism, it bei alleged that substances in milk other than formaldehyde produce colour reaction Many analysts prefer to use sulphuric acid for the test, but the latter is far le delicate, because of the charring that inevitably results by the action of the acid the milk. The reliability of the hydrochloric acid method has been tested again a again in this laboratory, and, while it may not be considered best to rely on this te as the sole evidence of formaldehyde for purposes of a court case, but rather employ it as a preliminary test, it can nevertheless be said that the writer has ne known of a single instance in which the presence of formaldehyde as indicated by t colour reaction with hydrochloric acid was not confirmed by subsequent tests, su for instance, as testing the first few drops of the distillate from the milk with Schiff's reagent, or with the mixture of resorcin and sulphuric acid recommended by Mulliken. Various aldehydes, when present in milk, give colour reactions when treated with hydrochloric acid as above, but nothing but formaldehyde gives the peculiar violet coloration which is perfectly distinguishable and unmistakable to the practised eye.

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* Samples examined for preservatives during July and August only.
† Samples examined for preservatives during June, July, August, and September.

The preliminary tests for boracic acid and carbonate require even less time than that for formaldehyde, being made in each case in the original platinum dish in which the 5-gramme portion for total solids was weighed out. In fact, the tests are so arranged as to be, as it were, only a side-issue in the process of cleaning the platinum dishes. The dishes are, of course, numbered, and the residues from the total solids are incinerated to an ash in each dish one after another. The dishes containing the milk ashes are then arranged side by side, and, by means of a pipette, 1 or 2 drops of dilute hydrochloric acid are introduced into each in succession, taking notice whether effervescence ensues, which in itself would indicate the presence of carbonate, and is very perceptible when the carbonate is present in as small amount as 0·05 per cent. In case of effervescence, the corresponding sample of the original milk is further examined by the rosolic acid test for carbonate.

Continuing, the test for boracic acid is carried out by adding with a wash-bottle a few cubic centimetres of water to the acidulated ash of each milk sample in the dishes, moving each dish slightly to get the ash into solution, and finally placing in each solution a strip of turmeric paper. Formerly it was customary to at once place these strips of turmeric paper on parallel glass rods for drying, the arrangement being such that each strip of paper occupied a numbered position corresponding to the number of the platinum dish from which it was taken; but of late it has been found simpler and just as efficient, having dipped each strip in the solution, to allow it to adhere to the side of the original dish, and remain there until dry. If the paper, when dry, is of a deep cherry-red colour, turning a dark olive when treated with a dilute solution of sodium hydroxide, the presence of boracic acid is assured.

These rapid methods, if carefully conducted to avoid confusion of samples, are serviceable in indicating at least, in a preliminary way, the samples containing added preservative, as well as the preservative employed, and in all cases as many confirmatory tests as desired may be conducted.

THE DETECTION OF STEMS IN GROUND CLOVES.

By Edgar B. Kenrick.

It would appear that the green colouring matter (chlorophyll) is absent from the clove-buds, while the stems contain it in considerable quantity. To detect stems—a common adulterant in ground cloves—the following simple method may be used: Shake up a small quantity of the material in a test-tube with alcohol, allow the sediment to settle, and examine the absorption spectrum of the tincture for the characteristic bands of chlorophyll. Nearly all samples of whole cloves contain, when purchased, a certain proportion of stems. These may be picked out, ground up, and used for purposes of comparison.

University of Manitoba, Winnipeg, *October* 3, 1901.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

Recognition of Boiled Milk. Kühnau. (*Milch Zeit.*, 1901, xxx., 327; throu *Chem. Zeit. Rep.*, 1901, 184.)—The author recommends Arnold's test with guaiac tincture for this purpose; but it is important that the light-brown tincture of guaiac wood be employed, not the resin tincture or an ammoniacal product. The tinctu of the wood should be tested before use: 1 part mixed with 15 of raw milk shou quickly yield a dark-blue colour which soon disappears, and comes again on addi more reagent. Boiled milk, or a sample heated above 80° C., only gives a di yellow colour. F. H. L.

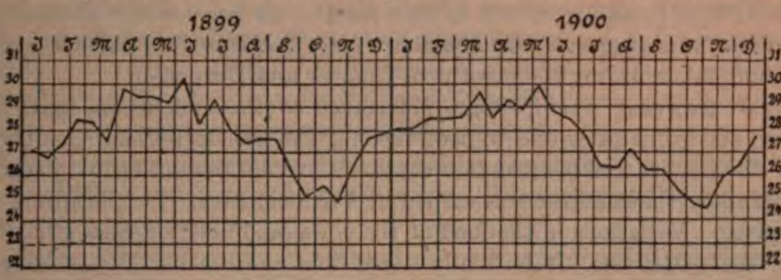
The Detection of Cocoanut Oil in Cocoa Butter and Chocolate. J. Waute (*Bull. de l'Assoc. Belge*, 1901, xv., 131, 132.)—This is readily effected by the autho distillation method (ANALYST, xxvi., 128), as is shown by the following results obtain with pure samples of the respective fats:

	Soluble Volatile Acids.			Insoluble Volatile Acids.		
	1st Distillation.	2nd Distillation.	Total.	1st Distillation.	2nd Distillation.	Total.
Cocoa butter ...	0·10	0	0·10	0·25	0·15	0·4
Cocoanut oil ..	7·10	4·30	11·40	7·85	7·55	15·4

C. A. M.

The Halphen Reaction and American Lard. P. Soltsien. (*Zeits. öffentl Chem.*, 1901, vii., 140; through *Chem. Zeit. Rep.*, 1901, 167.)—The author ha prepared samples of fat from American pigs which had been fed on cottonseed cake The material was soft and of a brownish-yellow colour. With the Halphen test i gave a colour suggesting the presence of more than 0·25 per cent. of cotton oil. Th nitric acid test gave even a stronger indication. Becchi's test also succeeded, thoug more faintly, but the Welman test for cotton oil failed. The iodine value of the fa was rather high, 68, and therefore inconclusive. Apparently the only proof that cotto oil had not been added to the lard was the fact that phytosterin was absent. Soltsie therefore considers that if a lard fails to give the Halphen reaction, less than 0·2 per cent. of cotton oil is present, and the sample need not be examined further; bu if the Halphen test succeed, phytosterin must be sought for. Probably the activ material in the Halphen and the nitric acid tests is identical. F. H. L.

The Reichert-Meissl Number for Butter. M. Siegfeld. (*Zeit. für Untersuch der Nahr. und Genussmittel*, 1901, 433; and **P. Vieth** (*Ber. Milchwirtschaft. Ins Hameln.*, 1900.)—The authors have carried out a considerable number of investig tions for the purpose of ascertaining the variations which the composition of butter undergoes during different times of the year. The butters from four North Hanover dairies were analysed twice a month during the whole of 1899 and 1900, and the



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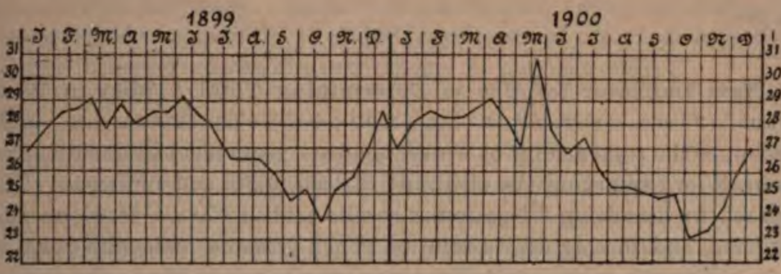

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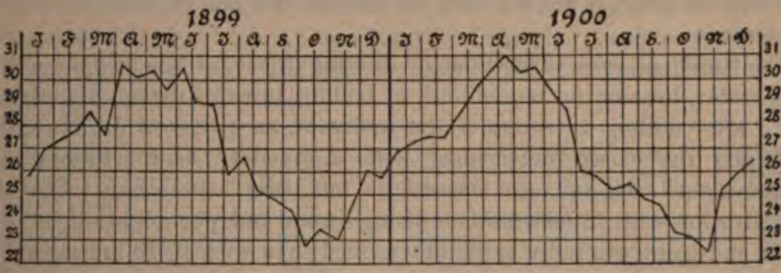

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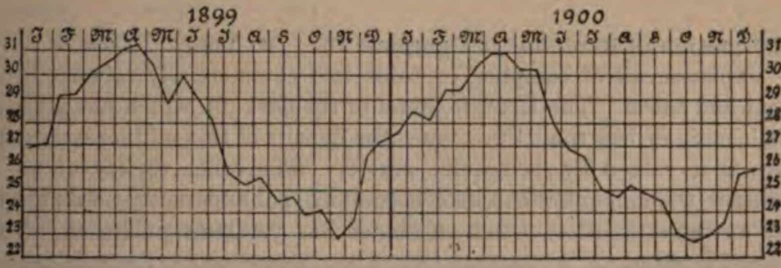


D

results given in the table show that the Reichert-Meissl number was highest (28 to 31·3) in the months of April, May and June, and lowest (22·4 to 26·4) in October and November of each year. The butters were from the mixed milk of about 5,000 cows.

The dairies from which they came were situated at Bülkau, Wesermarsch, Esens, and Georgsheil, and are designated A, B, C, D respectively in the tables. It will be noticed that the numbers obtained vary from 24·6 to 29·9 for A, 23·2 to 30·9 for B, 22·4 to 31·0 for C, and from 22·8 to 30·9 for D. In considering whether a butter is adulterated, it is suggested that the above facts be taken into consideration, a lower limit being taken during the months of October and November.

A. J. Swaving (*ibid.*, 1901, 577) comes to the conclusion that, in the late autumn, and principally due to poor feeding, the Reichert-Meissl number decreases whilst the Crismer number (the temperature at which the butter dissolves in absolute alcohol, under the conditions described by Crismer) rises. In the spring the reverse takes place. Variations in the food of the cows are quickly detected in the composition of the butter-fat. It is finally stated that the Reichert-Meissl number is not specially lowered in the autumn if the food of the cows be supplemented by feeding-cakes, etc.

W. P. S.

A New Indicator for Use in Determining Total Acidity in Wines. E. G. Runyan. (*Journ. Amer. Chem. Soc.*, xxiii., 402.)—The author has applied to the estimation of the total acidity of wines, vinegars, and ciders an indicator proposed by L. Lachaux for estimating the alkalinity of beet-juices and molasses. The indicator is prepared by dissolving 3·1 grammes rosolic acid in 150 c.c. of 90 per cent. alcohol, neutralizing, and mixing with a solution of 0·5 gramme malachite-green in 50 c.c. of alcohol. With this mixture alkalies give a purple colour, changed to green by acids. The malachite-green is not affected, but renders the end reaction of the rosolic acid more distinct.

The titrations were carried out by diluting 10 c.c. of the sample with 300 c.c. of boiling water, heating for one minute to boiling to expel carbon dioxide, and cooling to 75°; after adding 10 drops of the indicator, an excess of decinormal alkali was added, and the excess determined with a standard acid solution. The results obtained agreed well amongst themselves, but were lower than those obtained by direct titration with standard alkali, using litmus and phenolphthalein as indicators, and the end-point was more easily determined than with these indicators.

A. G. L.

Estimation of Camphor in Camphor Oil. H. Löhr. (*Chem. Zeit.*, 1901, xxv., 292.)—Japanese oil of camphor contains two commercially valuable constituents—camphor and safrol. The latter is found in the dark oil, which has a specific gravity of a little more or less than 1·0. Since the density of this oil increases with its safrol content, it is valued by its specific gravity. The other variety of oil is valuable according to the amount of camphor it contains, but apparently no rapid and accurate process for the estimation of that substance has yet been published. The camphor is obtained from the oil by exposing it to a low temperature, the solid which separates being removed. Then the oil is distilled, and the fractions containing the camphor are treated similarly until the whole of that material has been recovered. The author's analytical process is an imitation of this method on the small scale:

At least 300 grammes of the oil are separated by distillation into three fractions: (*a*) Up to 195° C.; (*b*) between 195° and 220°; and (*c*) above 220° C. The middle fraction is placed for an hour in a freezing mixture, and the camphor which crystallizes out is collected by means of the pump. It is packed in filter-cloth, wrapped up in paper, and pressed for half an hour, then clean paper is applied, and the pressure repeated for fifteen minutes. The oily liquor is again distilled similarly four times, cooling the fraction passing over between 205° and 220°, and pressing only once for twenty minutes. The camphor recovered is finally weighed. If the original sample is very viscid, and therefore contains a large quantity of camphor, it should first have part of that constituent removed by simple freezing. Various examples are quoted which appear reasonably accurate.

Löhr mentions that two specimens of camphor oil were recently examined in a municipal laboratory in Japan by distilling them and collecting the fractions 195° to 215° C., which solidified on cooling. These were returned as "camphor chiefly," and the samples were stated to contain 54·13 and 64·58 per cent. of camphor respectively, whereas by Löhr's process the same oils gave 19 and 21 per cent. only.

F. H. L.

Oils of Sandalwood, Lavender, and Thyme. L. F. Kebler. (*Amer. Journ. Pharm.*, 1901, lxxiii., 223-227.)—*Sandalwood Oil.*—The physical and chemical constants generally accepted for a pure oil, which must readily dissolve in 70 per cent. alcohol, are: Specific gravity at 15° C., 0·970 to 0·978; optical rotation at 25° C. in a 100-millimetre tube, $-17°$ to $-19°$; santalol, at least 90 per cent. Sample No. 1 in the subjoined table was prepared by the author from a wood yielding 5·5 per cent. of oil. Its constants fall well within these limits. The other samples were representative of the commercial oil. The author regards No. 6 as undoubtedly adulterated, and Nos. 3, 4, and 9 as inferior.

No.	Specific Gravity.		Santalol, Per Cent.	Optical Rotation.	Solubility in 70 Per Cent. Alcohol.	Santalol Esters, Per Cent.
	15° C.	25° C.				
1	0·9767	0·9724	97·16	$-17°15'$	1 in 5	3·06
2	0·9727	0·9707	93·64	$-18°16'$	,,	4·10
3	0·9747	0·9739	91·70	$-14°56'$	,,	2·93
4	0·9666	0·9601	90·12	—	,,	1·48
5	0·9716	0·9685	92·87	$-17°2'$	,,	1·43
6	0·9626	0·9600	75·00	$-7°4'$	,,	2·67
7	0·9721	0·9681	96·34	$-16°36'$	,,	—
8	0·9713	0·9678	94·53	$-16°56'$	,,	3·61
9	0·9734	0·9696	90·87	$-13°48'$	1 in 5½	—

Oil of Lavender.—According to Gildemeister and Hoffmann, oils containing less than 30 per cent. of esters are usually adulterated. In the author's opinion, however, this statement is too sweeping, for genuine oils of lavender are known containing as little as 10 per cent. of esters, and yet highly esteemed commercially.

The four examples examined by the author were all considered to be of good quality. They gave the following results:

No.	Specific Gravity at 15° C.	Solubility in 70 Per Cent. Alcohol.	Optical Rotation.	Esters, Per Cent.
1	0·8985	1 in 3	- 6°6′	25·70
2	0·8989	,,	- 2°54′	34·36
3	0·8892	,,	- 5°9′	31·42
4	0·8830	,,	- 3°41′	28·29

Oil of Thyme.—The author gives the following as the characteristics of the genuine oil: Soluble in 1 to 2 vols. of 80 per cent. alcohol; specific gravity at 15° C. 0·900 to 0·935; and amount of phenol bodies, 20 to 30 per cent.

He has obtained the following results with different commercial oils:

No.	Kind.	Specific Gravity at 15° C.	Solubility in 80 Per Cent. Alcohol.	Phenol Bodies, Per Cent.	Optical Rotation.
1	White	0·877	Insol. in 20 vols.	2·55	—
2	,,	0·881	Insol. in 20 vols.	4·26	—
3	,,	0·863	Insol. in 10 vols.	None	—
4	,,	0·8964	Sol. in 2 vols.	4	- 3°48′
5	,,	0·8935	Insol. in 10 vols.	27	- 3°48′
6	Red	0·907	Sol. in 2 vols.	25·56	- 1°24′
7	,,	0·880	Insol. in 10 vols.	8·73	—
8	,,	0·893	Insol. in 10 vols.	18·81	- 1°6′
9	,,	0·916	Sol. in 1¾ vols.	30·16	- 2°
10	,,	0·9231	Insol. in 10 vols.	19·00	—
11	,,	0·9084	Sol. in 2 vols.	14	+ 1°48′
12	,,	0·9074	Sol. in 2 vols.	24	- 1°30′

In the author's opinion, there is but little genuine oil of thyme on the market, the white oil in particular being largely adulterated with turpentine.

Of the red oils in the above table, Nos. 6 and 12 were considered genuine.

The proportion of phenol bodies was determined by shaking 10 c.c. of the oil with a 5 per cent. solution of sodium hydroxide in a 100 c.c. nitrometer for five minutes, and then leaving it for twenty-four hours. The amount of oil freed from phenol could then be read off and the percentage calculated. C. A. M.

Looff's Process for the Estimation of Morphine in Opium. N. A. Orlow and P. K. Horst. (*Farmaz. J.*, 1901, xl., 91; through *Chem. Zeit. Rep.*, 1901, 105.) —This process (ANALYST, 1896, xxi., 163) gives lower results than those propounded by Dieterich and Flückiger; and the present authors have accordingly investigated all three to ascertain which gives the purest product. The figures obtained were:

	Morphine.	Nitrogen in Morphine.
By theory	—	4·91 per cent.
Looff	10·21 per cent.	4·72 ,,
Dieterich	11·71 ,,	3·57 ,,
Flückiger	11·94 ,,	3·51 ,,

F. H. L.

ie **Assay of Conium Seed or Leaves.** H. M. Gordin. (*Amer. Journn.*
, 1901, lxxiii., 217, 218.)—Conium cannot be assayed by the author's general
(ANALYST, xxv., 74) owing to the facts that coniine is not completely precipi-
y Mayer's or Wagner's reagent, and that the alkaloid is so volatile, even at the
y temperature, that its solutions in immiscible solvents cannot be evaporated
loss.

e following modification of Cripp's method is said to give excellent results:
ımes of finely-powdered conium are shaken in a 300 c.c. stoppered bottle with
of a previously-prepared mixture of 1 part of chloroform and 3 parts of ether
ıt five minutes. Ten c.c. of a 10 per cent. solution of potassium hydroxide are
troduced, and the bottle shaken at intervals for four hours and set aside over
The next day 100 c.c. of the clear liquid are transferred, by means of a
into a 300 c.c. flask, and thoroughly mixed with 10 c.c. of a 2 per cent.
of oxalic acid in alcohol. The liquid is completely distilled off, the last traces
xpelled by blowing air through the flask, which is warmed on the water-bath.
oling, 10 c.c. of absolute alcohol are introduced, and the flask gently heated,
in cooled.

e alcoholic solution is now filtered into a wide beaker, and the flask washed
ı three successive portions of 5 c.c. of absolute alcohol. The beaker is placed
rm water-bath, and after almost complete evaporation of the alcohol, 10 c.c.
r are added, and the whole made up to 25 c.c., shaken with about 2 grammes
and filtered.

5 c.c. of the filtrate (= 5 grammes of the drug) are then shaken in a separating
vith 25 c.c. of petroleum spirit (boiling below 60° C.), and 5 c.c. of a 10 per
lution of potassium hydroxide. The lower layer is withdrawn and shaken
cond separating funnel with 20 c.c of petroleum spirit, the new lower layer
ff into a beaker, and the upper layer transferred to the first funnel. The
liquid in the beaker is again shaken with fresh quantities of petroleum
ntil the residual liquid gives no precipitate when rendered acid and tested
agner's reagent.

e united petroleum spirit extracts are shaken for fifteen minutes with about
nme of magnesium oxide, and then filtered into a 300 c.c. flask, the filter
vered with a watch-glass. The filtrate and washings are mixed with 50 c.c.
r saturated solution of hydrochloric acid gas in anhydrous ether, and the
vaporated on a water-bath, the last traces of the solvent and free acid being
by a current of dry air.

m 25 to 30 c.c. of $\frac{N}{10}$ silver nitrate solution and 5 c.c. of 10 per cent. nitric
now introduced into the flask, which is warmed on the water-bath until the
ecomes clear. The flask is then cooled, and its contents made up to 100 c.c.
ered. The chlorine in 50 c.c. of the filtrate is determined by Volhard's

number of c.c. of $\frac{N}{10}$ silver nitrate consumed by the 5 grammes of the drug,
ed by 0·0635, gives the percentage of coniine in the sample. C. A. M.

Uganda Aloes. A. Tschirch and J. Klaveness. (*Arch. Pharm.*, 1901, ccxxxi 241; through *Chem. Zeit. Rep.*, 1901, 177.)—The Uganda aloe has recently bee introduced into commerce. It belongs to the Cape aloe class, and its aloin is identic with Cape-aloin. Its resin is a compound of paracumaric acid with Uganda-resin tannol, which is identical with the resinotannol of Natal aloes. The latter has t composition $C_{22}H_{22}O_8$. The Uganda aloe also contains emodin. F. H. L.

Analysis of Fluid Extract of Condurango (*Gonolbus Condurango*). **J. Wari** (*Journ. Pharm. Chim.*, 1901, xiii., 506-512.)—This preparation is found in the Swi and German Pharmacopœias, but not in those of England or America. It prepared by macerating the powdered bark with a mixture of glycerin, alcohol a water, though in the author's opinion it would be advisable to replace the glyce by alcohol and so dissolve more of the resin, which is regarded as one of the acti principles.

For the analysis the author has worked out the following method: Ten c.c. the fluid extract are mixed with 40 c.c. of water, which is sufficient to precipita the whole of the resin. The liquid is then brought to the boiling-point to expel t alcohol. By this treatment the condurangine is also precipitated, but redissolves the liquid cools.

The precipitate of resin is collected on a weighed filter, washed with 100 c.c. water, dried and weighed. The filtrate is treated with 150 drops of a solution tannin (1 : 25), and the precipitated alkaloid collected on a weighed filter, dried a weighed.

The following results were obtained with extracts prepared with alcohol different degrees of strength, and with and without glycerin:

Preparation.	Strength of Alcohol, Per Cent.	Specific Gravity.		Residue Dried at 100° C.	Residue, Dry or with Glycerin.	Resin, Per Cent.	Weight o Tannin Precipitat Per Cent
		Fresh.	After 2 Months.				
Swiss ...	22·5	1·074	1·0585	23·37	Glycerin	0·22	1·46
German ...	22·5	1·072	1·0583	23·52	,,	0·25	1·80
,, (*a*)	30	1·0298	1·0297	15·85	Dry	0·38	2·20
,, (*b*)	45	1·0122	1·0121	17·27	,,	0·56	3·65
,, (*c*)	60	—	—	17·86	,,	1·43	3·28

C. A. M.

TOXICOLOGICAL ANALYSIS.

Spectroscopic Examination of Blood. B. Tollens. (*Ber.*, 1901, xxxiv., 1426 —When a blood solution is examined spectroscopically the two absorption bands (oxyhæmoglobin are quite distinct, but the single band of reduced hæmoglobin lacking in sharpness. If the blood is treated with a little formaldehyde, the ox hæmoglobin bands are not affected in the least, but if the liquid is then gently warm with ammonium sulphide they gradually vanish, and almost exactly midway betwe them appears a single sharp, black band. On cooling the solution and shaking it with air the single band disappears, and the pair become visible again; on warming once more the phenomena recur, and so on. In presence of carbon monoxide, formaldehyde does not produce this effect, as the two bands of carbon monoxide-hæmoglobin resist the action of the reagent. F H. L.

Recognition of Bromoform and Bromal in Cases of Poisoning. D. Vitali. (*Boll. Chim. farm.*, 1901, xl., 173; through *Chem. Zeit. Rep.*, 1901, 194.)—The material to be examined is brought with water into a flask and distilled in a current of hydrogen. The bromoform volatilizes and passes over with the gas, although its boiling-point is 151° C. If the gas is led through a tube narrowed at the extremity, ignited, and the flame allowed to play on copper gauze, the metal is coloured blue by the formation of copper bromide. If the products of combustion are drawn through weak ammonia in a Woulff's bottle and the liquid is acidified with nitric acid, the bromine may be determined with silver nitrate. If the stream of gas is led over a dry mixture of thymol and potassium hydroxide, the substance turns violet; and if an alcoholic solution of potash containing a little aniline is employed, the odour of isobenzonitrile may be detected. By causing the products of combustion to play on a basin moistened with ammonia, a white cloud of ammonium bromide is produced, which soon condenses to fine crystals.

Bromal may be recognised similarly. The material is first distilled, and the distillate is treated with potassium hydroxide, which breaks up the volatilized bromal into potassium formate and bromoform. The methods are analogous to those suggested in 1881 for the identification of chloroform and chloral. F. H. L.

ORGANIC ANALYSIS.

The Oxidation of Nitrogen as a Source of Error in the Estimation of Hydrogen and Methane. Alfred H. White. (*Journ. Amer. Chem. Soc.*, xxiii., 477.)—The author found that oxides of nitrogen are always formed in explosion analysis, the amount increasing with the violence of the explosion, and that the dilution recommended by Bunsen when working with cylindrical eudiometer tubes under reduced pressure is not sufficient when working with the spherical Hempel pipettes, in which, since the explosion is more rapid, and the walls take up less of the heat generated, the temperature reached is higher. If the explosive ratio (ratio of inert to explosive gas) is kept between three and four, the error is negligible in the technical analysis of gases consisting almost entirely of hydrogen, but increases with the quantity of methane present, and may amount to more than 1 per cent. The method of Dennis and Hopkins (*Journ. Amer. Chem. Soc.*, xxi., 398), in which the gas is burnt quietly in a current of air and oxygen by means of a glowing platinum spiral tends to give high results, and care must be taken that the wire is not heated too much. Apparently no method involving active combustion can give strictly accurate results. A. G. L.

Spectroscopic Detection of Methylfurfurol. K. Oshima and B. Tollens. (*Ber.*, 1901, xxxiv., 1425.)—Widtsoe and Tollens have already shown that methylfurfurol derived from the methylpentosans and methylpentoses of natural products gives a characteristic absorption band in the spectroscope. The present authors have discovered that an addition of phloroglucinol renders the test more delicate and noticeable. The organic matter is distilled with 1·06 hydrochloric acid. Five c.c. of the distillate are treated with an equal volume of strong hydrochloric acid, and a little of a solution of phloroglucinol in 1·06 HCl is added. A red precipitate of methylfurfurolphloroglucide falls, which is filtered off after five minutes' standing; the filtrate is coloured orange, and before the spectroscope shows a dark band at the beginning of the blue, while the violet is partially obscured. F. H. L.

The Detection and Estimation of Artificial (Cellulose) Silk in Fabrics. M. Duyk. (*Bull. de l'Assoc. Belge*, 1901, xv., 166-175.)—The fibres are first treated in the cold with a 5 per cent. solution of sodium hydroxide, to which are added a few drops of a solution of sodium hypochlorite, if required. They are then immersed in dilute hydrochloric acid (1 : 10), washed with water, pressed between filter-paper, and dried at a gentle heat. They are subjected to the following tests:

Combustion.—Artificial silk burns readily without emitting the odour given by animal fibres, and leaves a white ash.

Action of Sodium Hydroxide (2 *per cent. solution*).—Artificial silk is scarcely attacked, whereas natural animal fibres are dissolved more or less readily.

Action of Concentrated Sodium Hydroxide.—When immersed in a solution of specific gravity 1·33, fibres derived from cellulose are converted into a gelatinous mass.

Action of Nitric Acid.—Natural animal fibres are coloured yellow through the formation of xanthoproteic acid, whilst artificial silk is decomposed and frequently falls to powder.

Millon's Reagent.—On immersing the decolorized fibres in the boiling reagent, artificial silk does not alter, whereas natural silk acquires a deep red colour and wool a reddish-yellow colour.

Pyroxyline Test.—The following test is prescribed by the French Customs authorities: A little of the material is freed from any dressing, dried, and immersed in a mixture of 2 parts of sulphuric acid (specific gravity 1·835) and 1 part of nitric acid (specific gravity 1·50) at a temperature of about 25° C., and left for an hour. Natural silk is dissolved, whilst artificial silk is nitrated. In the latter event the fibres are thoroughly washed, dried, and identified by their behaviour on ignition and solubility in acetone.

In the author's experience this method is not altogether satisfactory, for it does not distinguish between artificial silk and other fibres of vegetable origin.

Ammoniacal Solution of Nickel Carbonate.—This dissolves the fibres of natural silk, but is practically without action on other animal or vegetable fibres. The reagent is prepared by dissolving 1 part of nickel carbonate in 6 parts of ammonium hydroxide, and diluting the solution with 6 parts of water. The fibre is immersed for a few minutes in the cold liquid. After some time wool, too, is slightly attacked.

Concentrated Sulphuric Acid.—Artificial silk is dissolved. Chardonnet's artificial silk when thus dissolved gives the reaction for nitrates with diphenylamine sulphate.

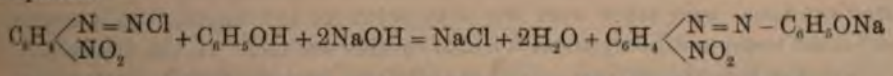
Microscopical Examination.—Apart from characteristic differences in the appearance of the different fibres, which are illustrated by figures, the author recommends the following micro-chemical tests:

> *Iodine Water and Dilute Sulphuric Acid.*—Natural silk is coloured yellow, whilst artificial silk assumes a deep blue colour. Other vegetable fibres take various colorations, from bright blue (cotton, flax) to yellowish-red (jute, etc.).
>
> *Ammoniacal Solution of Copper.*—This dissolves cellulose-silk almost instantly, apparently without first causing it to swell up, as in the case of natural vegetable fibres, which are covered with a ligneous cuticle.
>
> *Aniline Hydrochloride.*—A 2 per cent. solution of this salt gives a yellow or brown coloration with natural vegetable fibres, but not with artificial cellulose-silk. The fibre of *asclepias* (vegetable silk) acquires an intense yellow colour under these conditions.

Quantitative Analysis.—A weighed quantity of the material is freed from dressing and immersed in the author's ammoniacal nickel reagent. The blue solution is rapidly decolorized, and if necessary more must be added. After thirty minutes the fibres are washed with water, then with water slightly acidulated, and again with water, and dried at 105° C., and weighed.

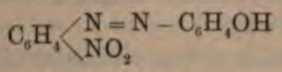
When wool is present the older method of boiling with a 2 per cent. solution of sodium hydroxide is employed. C. A. M.

A New Method of Estimating Phenols. E. Riegler. (*Bull. Soc. Bucuresci*, viii., 51; through *Ann. de Chim. anal.*, 1901, vi., 231.)—This is based on the property possessed by phenols of forming a colouring matter with para-diazo-nitraniline, as in the equation



$$C_6H_4\left\langle\begin{matrix}N=NCl\\NO_2\end{matrix}\right. + C_6H_5OH + 2NaOH = NaCl + 2H_2O + C_6H_4\left\langle\begin{matrix}N=N-C_6H_5ONa\\NO_2\end{matrix}\right.$$

On adding sulphuric acid drop by drop to the alkaline solution, the colouring matter separates out in the form of the compound



$$C_6H_4\left\langle\begin{matrix}N=N-C_6H_4OH\\NO_2\end{matrix}\right.$$

This substance, which is nearly insoluble in water, is collected on a filter and weighed. The weight multiplied by the factor 0·3868 gives the corresponding amount of phenol. As a correction for the slight solubility 0·002 gramme is added to the weight found, for each 100 c.c. of liquid used. The author has used the same method with satisfactory results in the determination of thymol and guaiacol. C. A. M.

On the Elimination and Quantitative Estimation of Water in Oils, Fats, and Waxes. Charles B. Davis. (*Journ. Amer. Chem. Soc.*, xxiii., 487.)—The drying may be very readily and safely effected as follows: Into a wide-mouthed weighing bottle sufficient thick filter-paper (in coil form) is introduced to half fill the vessel. Bottle and paper are then dried at 110° C. to constant weight, and as muc of the sample is added as will just saturate the filter-paper. The increase in weigh of the bottle gives the quantity taken. The whole is then dried at 110° C., if neces sary in an atmosphere of carbon dioxide or hydrogen, and again weighed, the los giving the water evaporated.

In some cases it may be more convenient to introduce the oil in ethereal solution, in which case the ether is first evaporated off at a slightly elevated temperature, and the sample then dried as above. Solid fats and waxes are added in their natural state, and are quickly absorbed by the paper after melting. The substance is easily recovered by extracting the paper in a Soxhlet apparatus. A. G. L.

A Siccative for Oil-colours in Tubes. Moriz Kitt. (*Chem. Revue über d. Fett-u-Harz-Ind.*, viii., 157.)—It has been customary for a long time to add 5 to 10 per cent. of wax to many oil-colours as a thickening agent, in order to prevent the heavier pigments from separating out. Several objections are urged by painters against the use of wax for this purpose, and a material more suitable appears to consist in cold-drawn wood oil, which has turned solid through exposure to light for a few weeks. A number of experiments were made which show that wood oil ha the further advantage of causing the colour to dry more quickly. A. G. L.

The Separation of Cholesterin and Phytosterin from Mixtures of Fatty an Mineral Oils. J. Marcusson. (*Chem. Revue über d. Fett-u-Harz-Ind.*, 1901, viii., 10 105.)—In the case of thin mineral oils which are volatile with superheated steam th following method has given satisfactory results in the author's hands: From 100 200 grammes of the oil are saponified with alcoholic alkali, and the unsaponifiab matter extracted with ether from the soap solution, and distilled in a current superheated steam until only about 3 c.c. of residue was left. In the distillation ca must be taken that the temperature of the oil never exceeds 200° C.

The residue from the distillation is boiled with 70 per cent. alcohol, and th crystals of cholesterin or phytosterin obtained from the extract are purified b recrystallization in the usual way.

When the mixture contains a heavy mineral oil, the preceding method is n applicable, and in such cases the following method is recommended: The oil unde examination is dissolved in ether, and the greater proportion of the mineral c precipitated by the addition of an equal volume of alcohol. The clear supernata liquid is poured off, concentrated to about 50 c.c., and, after cooling, again decante from any further deposit, and evaporated. The residue is saponified, and th aqueous soap solution extracted several times with ether. The residue from th extract is then crystallized from 70 per cent. alcohol, as in the first method.

C. A. M.

INORGANIC ANALYSIS.

Examination of Commercial Copper Oxide. **D. Miklosich.** (*Zeits. f. angew. Chem.*, 1901, 753.)—Commercial cupric oxide is liable to contain cuprous oxide, which greatly diminishes its value for colouring glass green. For the estimation of this impurity advantage may be taken of the reaction :

$$Cu_2O + Fe_2O_3 = 2CuO + 2FeO.$$

One gramme of ferrous ammonium sulphate is weighed out, dissolved in dilute sulphuric acid, and titrated with permanganate. Thus, the iron is all converted to the ferric state, and the strength of the permanganate solution is simultaneously determined. Ten c.c. of dilute sulphuric acid are then added, and also ½ gramme of the copper oxide, which is brought into solution by gently warming, this operation being carried out in a flask provided with a Bunsen valve to prevent oxidation. When cold the contents are transferred to a porcelain basin containing a litre of air-free water, and rapidly titrated with the permanganate solution.

Volumetric Estimation of Lead Peroxide in Red-Lead. **M. Liebig.** (*Zeits. f. angew. Chem.*, 1901, 828.)—The red-lead is reduced to a fine state of division, and 0·5 gramme is washed into an Erlenmeyer flask with a little distilled water. To this 25 c.c. of $\frac{N}{10}$ sodium thiosulphate are added, then 10 c.c. of approximately 30 per cent. acetic acid, and dissolution is brought about by shaking. Then 10 c.c. of potassium iodide solution (1 : 10) are added, and 2 to 3 c.c. of zinc iodide and starch solution, and the excess of thiosulphate is titrated with $\frac{N}{10}$ iodine solution. The number of c.c. of the thiosulphate solution oxidized by the red-lead, multiplied by 239, the molecular weight of the peroxide, gives the percentage of the latter. The end of the reaction is shown by the precipitated yellow lead iodide appearing dirty yellow.

A. M.

Iodometric Estimation of Antimonic Acid. Volumetric Determination of Antimony in Presence of Tin. **M. Rohmer.** (*Ber.*, 1901, xxxiv., 1565.)—Although antimonic acid is not quantitatively reduced by sulphurous acid at the boiling-point, the reaction succeeds in the presence of hydrobromic acid. Based on this fact, the following process has been elaborated : The original solution, containing antimonic and hydrochloric acids, is boiled in a round-bottomed flask with 1 gramme of potassium bromide, some aqueous sulphurous acid, and a few pieces of pumice, till the odour of SO_2 has disappeared. The liquid is cooled, treated with tartaric acid, neutralized with sodium bicarbonate, and titrated with $\frac{N}{20}$ iodine. This is best effected by adding about 1 c.c. more than the necessary quantity, and titrating back with thiosulphate of equivalent strength.

Tin hinders the oxidation of antimonous to antimonic acid, but that effect is overcome by the addition of large amounts of tartaric acid. Thus, a mixture of antimony and tin can be satisfactorily separated in the following manner : The mixed sulphides which have been obtained in the course of an ordinary analysis are brought into a round-bottomed flask, and boiled with strong hydrochloric acid and potassium chlorate till the odour of chlorine is no longer noticeable. One gramme of potassium bromide and some sulphurous acid are introduced, and the excess of the latter is boiled off. After cooling, a considerable quantity (10 to 20 grammes) of tartaric acid is added, and the liquid titrated as above. Alloys may be dissolved directly in aqua regia or in hydrochloric acid and chlorate. Antimony-tin-arsenic alloys are first treated with the latter solvent as usual, then bromide is added, and the arsenic is driven off by distillation in a current of sulphur dioxide and hydrochloric acid. F. H. L.

Modified Williams Method for Manganese. Randolph Bolling. (*Journ. Amer. Chem. Soc.*, xxiii., 493.)—In the following modification of the Williams' method, the time necessary for the filtration of the manganese dioxide is greatly shortened by the preliminary removal of the silica: 5 grammes of pig-iron drillings are treated in a beaker with 75 c.c. nitric acid (specific gravity 1·42), and, after violent action has ceased, 10 c.c. hydrofluoric acid are added. The solution is evaporated till ferric oxide begins to separate out, cooled, and heated to boiling after the addition of 100 c.c. nitric acid (specific gravity 1·42). Some asbestos fibre and then about 7 grammes potassium chlorate are added, and the solution is boiled until the green fumes have disappeared, cooled rapidly, and filtered through asbestos. After two washings with nitric acid, the precipitate is washed with cold water until the washings are neutral to litmus. The asbestos pad and precipitate are then transferred to the original beaker, 50 c.c. of standard acid ferrous sulphate solution run in, and after the precipitate is entirely dissolved the excess of ferrous sulphate is titrated with decinormal permanganate solution.

The solutions should be standardized by treating a sample of pig-iron with a known percentage of manganese as above, and, after the titration is finished, running in a second quantity of 50 c.c. of the iron solution, and titrating this with the permanganate. By this method of standardization the errors due to the asbestos and carbon floating about in the liquid, as well as those due to irregularity in the precipitate, are avoided. Results obtained by this method agreed very well with those obtained by the Gibbs' method, in which the manganese is weighed as pyrophosphate, the largest deviation being 0·02 per cent. A. G. L.

The Volumetric Determination of Zinc. Percy H. Walker. (*Journ. Amer. Chem. Soc.*, xxiii., 468.)—The following method is an application of Handy's modification of Stolba's method for the estimation of magnesium (*Journ. Amer. Chem. Soc.*, xxii., 31) to the case of zinc: To the zinc solution, which must contain ammonium chloride, a large excess of ammonia is added, and then a large excess of sodium phosphate. No precipitate is formed, but on cautiously neutralizing the excess of ammonia with dilute acid, the liquid gradually becomes milky, when it should be heated to 75° C., and the neutralization continued, with continuous stirring, until a piece of litmus-paper placed in the solution just turns violet, whilst there is no smell of ammonia. The crystalline precipitate is filtered after five minutes' standing and washed with cold water; paper and precipitate are returned to the beaker in which precipitation was effected, an excess of standard acid and a few drops of methyl orange added, and the excess is determined by standard alkali. According to the equation,

$$Zn(NH_4)PO_4 + H_2SO_4 = ZnSO_4 + (NH_4)H_2PO_4,$$

1 c.c. of normal acid corresponds to 32·7 milligrammes of zinc.

In three determinations by this method the average error was $-0·0006$ gramme on 0·1490 gramme zinc taken. The method may be used in the presence of magnesium, ferric iron, and calcium, since the phosphates of these three metals are precipitated in a strongly ammoniacal solution, and can be removed by filtration before neutralizing the liquid. In two determinations, in which all three metals were present, the error was $-0·0020$ gramme on 0·1192 gramme zinc taken. Manganese, if present, must be previously separated. A. G. L.

Determination of Zinc in Spathic Iron Ore. J. Flath. (*Chem. Zeit.*, 1901, xxv., 564.)—This process is specially suitable for the determination of small quantities of zinc (0·4 to 5·0 per cent.) in presence of much iron and manganese. Three to 5 grammes of material are dissolved by gentle warming in hydrochloric acid, diluted with 150 or 200 c.c. of water, and treated with a small excess of ammonia. Without filtering, the precipitate is dissolved in 15 c.c. of 96 per cent. acetic acid, and sulphuretted hydrogen is passed through the liquid to throw down all zinc, lead, copper, and 2 or 4 per cent. of the iron. This precipitate settles very quickly, which zinc sulphide alone does not; and, by avoiding access of air as far as practicable, it can be filtered in five or ten minutes' time. The filtrate is perfectly free from zinc: it always becomes milky, but from presence of sulphur only. The mixed sulphides are washed twice with sulphuretted hydrogen water, dissolved in aqua regia, evaporated with 10 c.c. of 1 : 1 sulphuric acid, diluted with 100 c.c. of water, boiled, and the liquid treated with 10 c.c. of 10 per cent. sodium thiosulphate to remove the copper. Five c.c. of nitric acid are added to the filtrate in order to destroy the excess of thiosulphate and to oxidise the iron, and the whole is somewhat concentrated. The manganese is next thrown down by two precipitations with bromine and ammonia, and in the filtrate the zinc is estimated volumetrically.

The foregoing method has the advantage of permitting 3 or 4 grammes of substance to be analysed without danger of losing zinc in the usual iron precipitate. The tedious and complicated precipitation of iron and manganese together is also wholly avoided. It yields higher results in zinc than the ordinary processes; and experiments on mixtures of spathic iron ore (free from zinc) with known quantities of a pure zinc salt show it to be more accurate. F. H. L.

On the Separation of Tungstic and Silicic Acids. H. L. Wells and F. J. Metzger. (*Journ. Amer. Chem. Soc.*, xxiii., 356.)—Otto Herting has recently stated (*Zeits. angew. Chem.*, 1901, 165) that the well-known method of expelling silica from tungstic acid by means of hydrofluoric acid is incorrect, since a part of the tungstic acid also volatilizes. The authors have examined the behaviour of mixtures of pure silica and tungstic acid when treated, before and after ignition over a Bunsen burner, with a mixture of hydrofluoric and sulphuric acids, and with hydrofluoric acid alone, followed by evaporation to dryness and ignition of the residue over a Bunsen burner. In all cases the silica was expelled quantitatively, the greatest alteration in the weight of the tungstic acid taken being 0·0001 gramme. The method thus appears to be perfectly exact, and the authors ascribe Herting's results to the fact that tungstic acid itself volatilizes slowly when ignited over the blast-lamp, and that he probably ignited his tungstic acid at too high a temperature.

A. G. L.

The Detection of Selenium in Sulphuric Acid. A. Jouve. (*Bull. Soc. Chim.*, 1901, xxv., 489-491.)—After referring to the codeine and sulphurous acid tests for selenious acid, the author points out that selenic acid is only slowly acted upon by sulphur dioxide, and does not give the codeine reaction. He finds that crude acetylene gives more or less pronounced red coloration when passed into sulphuric acid containing selenium, and that it will detect 1 part in 100,000.

The reaction is partially due to the reduction of the selenium compounds by impurities in the crude acetylene, although the pure gas is the principal agent. The speed of the coloration is increased by the presence of hydrochloric acid vapours.

C. A. M.

The Estimation of Carbonic Acid in Water. Joseph W. Ellms and Jay C. Beneker. (*Journ. Amer. Chem. Soc.*, xxiii., 405.)—As the result of an investigation into the comparative merits of the Pettenkofer, Trillich's modified Pettenkofer, and the Lunge-Trillich or Seyler methods (ANALYST, xxii., 312), the authors conclude that the last is the most accurate and rapid, the error on the "free and half-bound carbonic acid" being on an average less than 1 per cent., with a possible range of less than ±3 per cent. In titrating the "fixed" carbonic acid according to Hehner's method, the authors prefer the use of lacmoid to methyl orange, which is used by Seyler. Trillich's modification of Pettenkofer's method gives results which are generally 5 per cent., and may be as much as 12 per cent., too low. Pettenkofer's method gives still lower and less uniform values, and under some conditions it yields extremely erratic results.

A. G. L.

The Detection and Estimation of Nitrates in Water. P. Cazeneuve and H. Défournel. (*Bull. Soc. Chim.*, 1901, xxv., 639, 640.)—The authors' method is based upon the fact that a blood-red coloration is produced when a dry mixture of a nitrate and brucine is treated with glacial formic acid.

The residue obtained from the evaporation of a litre of the water is taken up with 20 c.c. of water and the solution evaporated in a small flat-bottomed dish on the water-bath with 0·05 gramme of brucine. A few drops of glacial formic acid are then added to the warm residue, and immediately afterwards a little water. A yellow tint, changing after twelve hours to rose, is obtained in the presence of a nitrate. The addition of hydrogen peroxide effects the change to rose in fifteen minutes. The limit of sensitiveness of the reaction is 1 : 100,000.

Unlike the colour produced by sulphuric acid and brucine with nitrates, which is fugitive, the rose coloration in this test remains unchanged for several days.

The test is made quantitative by comparing the colour with that obtained with a dry residue containing a known quantity of nitrate. C. A. M.

REVIEW.

HANDBOOK ON PETROLEUM. By Captain J. H. THOMSON and BOVERTON REDWOOD. London: Chas. Griffin and Co., Ltd. Price 8s. 6d. net.

The authors have done excellent service in writing this handbook, which contains in moderate compass and in very clear language a mass of practical information concerning petroleum, calcium carbide, and acetylene, and the precautions necessary for their safe conveyance, storage, and employment, which will be valuable to all who have to deal with these products, whether as inspectors under the Petroleum Acts, traders, or users. At the outset the authors explain that the legal definition of "petroleum" is a wide one, embracing many liquids and manufactured products which are not petroleum in the commercial sense; at the same time, the bulk of manufactured petroleum is not included in it, but only such as flashes below 73° F. They then give a brief history of petroleum, and a useful epitome of the theories concerning it, the most probable being that it is derived from organic remains, partly animal and partly vegetable.

Chapters II. and III. contain an account of the sources of supply and their relative importance; a detailed description of the petroleum well and its construction (which suffers in clearness owing to the absence of illustrations); the means employed for transport, storage, and distribution; and an outline of the refining processes. To supply the world's present consumption of petroleum, a pipe 41 inches in diameter would be required, assuming the rate of flow to be 3 feet per second.

In Chapter IV. the various commercial products obtained from petroleum, shale oil, and coal-tar are described, with their names and uses, specific gravities, flash-points, and boiling-points. This chapter will be useful to many, as there is a good deal of confusion respecting the precise significance of some of these commercial names. In the next chapter the meaning of "flash-point" and "fire-test" are explained in the clearest possible manner, and the conditions which influence the flash-point are discussed in detail.

With regard to the much-debated question whether the present legal flash-point of 73° F. needs alteration, the authors deprecate the basing of conclusions upon such considerations as the maximum atmospheric temperature, or the maximum temperature of the oil in any lamp, because the conditions of the test are not the same as those which arise in actual practice. They consider that the standard of flash-point should be judged by experience alone, and that no alteration should be made on merely theoretical grounds. "The real question is whether oil of a given flash-point has shown itself to be so dangerous by reason of its volatility as to require the standard to be raised, and, if so, whether the advantage in point of safety outweighs any disadvantage which may be involved." Statistics are quoted to show that the number of fatal lamp-accidents is not so great as to call for legislation of a very drastic character; in fact, more than five times as many fatalities are caused by people falling downstairs. If the flash-point were to be raised above the highest temperature which may occur in an ordinary lamp, it would have to be raised to at least 150° F. The British public, however, could not be compelled to use oil of this high test, and if oil of lower flash-point were found to be as good or better, and cheaper, it would continue to be used. The authors evidently consider any alteration of the existing flash-point both unnecessary and undesirable. They consider that 90 per cent. of the accidents which have occurred have been due to ignorance, carelessness, slovenliness, or false economy in purchasing flimsy lamps, and they look for the remedy to a better education of the people.

In Chapter VI. methods for determining the flash-point are detailed, with a history of the evolution of the Abel test. The Abel-Pensky test is very fully illustrated and described, "as advantage will probably be taken of the first opportunity to legalize its use in this country." The German instructions are given in full; also a table for correction of observed flashing-points for variations in barometric pressure, which, though not yet legalized, it is suggested that inspectors should make use of.

The bungling history of petroleum legislation is recounted in Chapter VII. In a fortunately abortive Bill introduced in 1869 nitroglycerin was included, "to which, apparently, the officer of the local authority was called upon to apply the flash-test"! The existing legislation is summarized in Chapter VIII., and the directions in which amendment and further legislation are desirable are pointed out.

In Chapters IX. and X. the precautions necessary to avoid accident in the conveyance, storage, and use of petroleum are fully discussed, and valuable suggestions are given as to the construction and use of mineral oil lamps, based upon the evidence given before the Select Committee on Petroleum. The last chapter is devoted to calcium carbide and acetylene. Sixteen appendices, containing the Petroleum Acts, Orders in Council, Model Rules for the Care and Use of Petroleum Lamps, etc., and an index complete the work.

It is to be hoped that the authors' wish may be realized, and that the general voluntary adoption of the safeguards which they have shown to be necessary may anticipate and even render unnecessary further legislation. To this end their handbook cannot be too widely read, and it should be possessed by all who have to advise professionally on matters relating to petroleum. L. A.

INSTITUTE OF CHEMISTRY.

We are informed that the next Examination in Therapeutics, Pharmacology, and Microscopy will be held at 30, Bloomsbury Square, London, W.C., in January, 1902, also that the exact date is not yet fixed, but will be communicated to intending candidates. Candidates desirous of presenting themselves are required to send in their applications on or before Tuesday, the 3rd day of December, 1901.

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THE ANALYST.

DECEMBER, 1901.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE monthly meeting of the Society was held on Wednesday evening, November 6, in the Chemical Society's Rooms, Burlington House.

The President (Dr. J. AUGUSTUS VOELCKER, M.A., B.Sc.) occupied the chair.

The minutes of the previous meeting were read and confirmed.

Certificates of proposal for election to membership in favour of the following candidates were read for the first time: Mr. Horatio Ballantyne, F.I.C., Analytical and Consulting Chemist, 75, Chancery Lane, W.C.; Mr. M. Hunter, M.A. (Oxon.), Science Lecturer, Rangoon College; Mr. Ernest Alfred Lewis, Assayer and Metallurgist to Muntz's Metal Co., Ltd., French Walls, near Birmingham; Mr. Frederick J. Lloyd, F.I.C., Analytical and Consulting Chemist, Muscovy House, Trinity Square, E.C.; Mr. Albert E. Parkes, F.I.C., Analyst to the Stepney Borough Council, 43, Whitehorse Street, Stepney, E.; and Mr. Thomas Tickle, B.Sc. (London), Public Analyst for the City of Exeter, 4, Hills Court, Exeter.

Mr. F. M. Wharton, F.I.C., was elected a member of the Society.

The following papers were read: "The Composition of Milk," by H. Droop Richmond; "The Determination of Carbon in Steel by Direct Combustion," by Bertram Blount; and "*Enteritidis sporogenes* as Evidence of Sewage Pollution," by M. Dechan.

The PRESIDENT (Dr. Voelcker) said that the Council had been requested to lay before the Departmental Committee on Butter Regulations appointed by the Board of Agriculture the views of the Society on the question of the composition of butter, the point dealt with by the Departmental Committee in the first instance being that of the maximum proportion of water that butter should contain. With the view of eliciting the opinions of members and putting forward an authoritative view on the question, there were circulated among the members of the Society forms of inquiry, the replies to which were collected and considered by a committee appointed by the Council for the purpose. These replies formed the basis of the conclusions which Dr. Dyer and Mr. Hehner, as the representatives of the Society, laid before the Departmental Committee in the course of their evidence. It was intended that the work of the Departmental Committee should include the consideration of questions relating not only to the proportion of water in butter, but also to the general nature and composition of butter, and this matter would form the subject of investigation and consideration by the committee appointed by the Society's Council, and of further evidence by their representatives.

Dr. DYER said that over ninety members of the Society had communicated their views on the question of water in butter. Of these a majority were of opinion that 16 per cent. should be fixed as a limit, and of the remainder a large preponderance were in favour of a lower limit, namely, 15 per cent., while some few recommended even lower limits still. Only a very few were in favour of a higher limit than 16 per cent. The committee appointed by the Council of the Society, having carefully weighed all this evidence, considered that it would on the whole express well the feeling of the Society if Mr. Hehner and himself were instructed to lay before the Departmental Committee, as the view prevailing in the Society, that the proportion of water permissible in butter should be fixed at 16 per cent., and a recommendation to that effect was accordingly made by them; but at the same time the diverse views of the minority of the members were also laid before the Committee. They were accorded a long and patient hearing, and underwent a long cross-examination on the subjects mentioned in the course of their evidence. He believed it was expected that the Departmental Committee would formulate some recommendation on the subject of water in butter in an interim report, without waiting until it had heard evidence on the much more difficult and knotty subject of the standards which should be fixed as indicative of genuine composition in the case of butter-fat.

THE COMPOSITION OF MILK.

BY H. DROOP RICHMOND, F.I.C.

(*Read at the Meeting, November* 6, 1901.)

Mean Composition during 1900.—Of the 34,978 samples examined in the Aylesbury Dairy Company's Laboratory during 1900, 29,482 were samples of milk. The composition of 13,798 received from the farms is given in Table I.

TABLE I.

Average Composition of Milk during 1900.

Month.	Morning Milk.				Evening Milk.				Average.			
	Specific Gravity.	Total Solids.	Fat.	Solids-not-fat.	Specific Gravity.	Total Solids.	Fat.	Solids-not-fat.	Specific Gravity.	Total Solids	Fat.	Solids-not-fat.
January ...	1·0326	12·48	3·49	8·99	1·0325	12·83	3·80	9·03	1·0326	12·66	3·65	9·01
February...	1·0325	12·45	3·48	8·97	1·0324	12·79	3·78	9·01	1·0325	12·62	3·63	8·99
March ...	1·0326	12·40	3·43	8·97	1·0325	12·77	3·76	9·01	1·0326	12·58	3·59	8·99
April ...	1·0324	12·33	3·40	8·93	1·0323	12·70	3·75	8·95	1·0324	12·52	3·59	8·93
May ...	1·0327	12·23	3·26	8·97	1·0325	12·66	3·67	8·99	1·0326	12·44	3·46	8·98
June ...	1·0326	12·21	3·27	8·94	1·0322	12·61	3·69	8·92	1·0324	12·41	3·48	8·93
July ...	1·0320	12·26	3·44	8·82	1·0314	12·43	3·69	8·74	1·0317	12·34	3·56	8·78
August ...	1·0318	12·26	3·47	8·79	1·0314	12·60	3·86	8·74	1·0316	12·43	3·66	8·77
September	1·0321	12·39	3·51	8·88	1·0317	12·70	3·88	8·82	1·0319	12·54	3·69	8·85
October ...	1·0323	12·56	3·62	8·94	1·0321	12·86	3·92	8·94	1·0322	12·71	3·77	8·94
November	1·0326	12·72	3·69	9·03	1·0323	12·98	3·98	9·00	1·0324	12·85	3·84	9·01
December	1·0327	12·63	3·61	9·02	1·0324	12·90	3·88	9·02	1·0325	12·76	3·74	9·02
Average ...	1·0324	12·41	3·47	8·94	1·0321	12·74	3·81	8·93	1·0323	12·57	3·64	8·93

The morning and evening milks have been kept separate. As has been observed in former years, the lowest fat occurs in May and June, and the highest during October and November. During July and August, as has been invariably the case in previous years, the solids-not-fat are lower than in the other months, and these begin to rise during September, and become normal towards the end of the month.

Accuracy of Methods Used.—As in the bulk of the analyses the fat is determined by the Gerber method and the solids-not-fat calculated, I have compared the results with those obtained by the Adams method for fat and the asbestos method for total solids.

In 78 analyses the difference between Adams and Gerber was:

In 69 cases, or 88·5 per cent., 0·1 or less;
,, 8 ,, 10·2 ,, 0·1 to 0·15; and
,, 1 case, or 1·3 ,, above 0·15.

The mean figures were identical.

In 82 analyses the difference between the fat calculated and that found was:

In 60 cases, or 73·2 per cent., 0·1 or less;
,, 19 ,, 23·2 ,, 0·1 to 0·15; and
,, 3 ,, 3·6 ,, 0·15 to 0·2.

The mean calculated fat was 0·019 per cent. less than the mean fat found.

It is evident that the figures in Table I. are close approximations to the truth.

Some experiments were made with Macfarlane's chrysotile method (ANALYST, xviii., 73). On shaking up a little chrysotile with water, it was found that the water was very distinctly alkaline to phenolphthalein, and it was thought that the lower results formerly obtained (ANALYST, xix., 77) might be due to heating the milk in the presence of soluble alkaline salts. A quantity of chrysotile was treated with strong hydrochloric acid, washed till neutral, and dried; a quantity of asbestos was similarly treated.

The results on one sample of milk were:

Dried on washed asbestos	12·70	per cent.	total solids.
,, unwashed asbestos	12·70	,,	,,
,, washed chrysotile	12·47	,,	,,
,, unwashed chrysotile ...	12·34	,,	,,

It was evident from these figures that the lower results are not wholly due, though perhaps partially, to the presence of alkaline salts.

The fat was estimated by extracting the dried solids with ether, with the following results:

Extracted from washed chrysotile	3·65	per cent.
,, unwashed chrysotile	3·62	,,
Adams's method	3·65	,,

The slightly lower result from unwashed chrysotile suggests that a small portion of the fat was saponified by the alkali; as the method, even after the very tedious process of completely washing the chrysotile was adopted, failed to give results of a satisfactory nature, it was not further investigated.

Variation in Fat during Delivery of the Milk.—The same average percentage of fat (3·64) was found in 7,359 samples taken before delivery and in 4,543 samples taken during delivery, though the variations were somewhat wider in the latter series than in the former, due to the tendency that cream has to separate from milk on standing. I have been asked what may be considered an extreme reasonable limit of variation, and the two series afford data for deducing this. I consider that the following formula gives a figure which is as correct as possible :

$$\text{Limit of variation} = 2{\cdot}022\sqrt{\frac{\Sigma^2_{,}}{n_{,}-1} - \frac{\Sigma^2_{,,}}{n_{,,}-1}}$$

where $\Sigma^2_{,}$ = the sum of the squares of the differences from the mean of the first series ;
$\Sigma^2_{,,}$ = the sum of the squares of the differences from the mean of the second series ;
$n_{,}$ = the number of samples in the first series ;
$n_{,,}$ = the number of samples in the second series.

The figure obtained for this value from the two series mentioned above is 0·31 per cent. ; this includes differences due to errors of sampling and analysis, and a reasonable amount of separation of cream ; the probable variation is only 0·11 per cent., and a higher difference than 0·31 per cent. would show either a very abnormal condition of things (*cf.* Vieth, ANALYST, viii., 2) or unnecessary carelessness.

Variation of Constituents of Solids-not-fat.—In order to ascertain whether all the constituents of the solids-not-fat vary directly as the total percentage, or whether an excess or deficiency of solids-not-fat is due to excess or deficiency of any one constituent, I have examined a large number of analyses of milk in which milk-sugar, proteids, and ash were determined. On plotting out the average figures for solids-not-fat against each of the constituents, I find that the figures for milk-sugar, proteids, and ash, lie each in a series of three straight lines. For each constituent the breaks occur between 8·8 per cent. and 8·9 per cent, and between 8·4 per cent. and 8·5 per cent., and are quite well defined. It is suggestive that the one figure is very near the average percentage, and the other is almost that adopted as the limit for normal milk, and the figures show that it would be difficult to dilute down a milk high in solids-not-fat without arousing a strong suspicion that it is watered. Table II. gives the figures deduced, which are averages, and individual samples may show differences.

TABLE II.

Solids-not-fat.		Milk-sugar.	Proteids.	Ash.
Range.	Average.			
about 10 per cent.	10·0	4·79	4·37	0·84
9·00—9·25	9·10	4·77	3·57	0·76
8·75—9·00	8·87	4·75	3·39	0·73
8·60—8·75	8·67	4·60	3·35	0·73
8·40—8·60	8·50	4·48	3·30	0·72
8·20—8·40	8·30	4·18	3·39	0·73
8·00—8·20	8·10	3·94	3·41	0·75

Any deficiency of solids-not-fat below 9·0 per cent. is chiefly due to a deficiency in the milk-sugar.

Any excess of solids-not-fat above 9·0 per cent. is chiefly due to an excess of proteids.

The ash may be deduced with very fair accuracy from the proteids by the formula A = 0·36 + 0·11 P.

Relation between the Proteids and Salts of Milk.—The formula just quoted indicates an intimate connection between proteids and salts of milk; it is, indeed, a matter of common knowledge that the casein is in some way combined with calcium phosphate, but so far as I am aware no attempt has been made to ascertain the exact way in which calcium phosphate is combined.

Following Söldner and Duclaux, I employed Zahn's method of filtration of the milk through a porcelain filter in my experiments on this point. It is usually stated, and I have hitherto accepted this statement, that on filtration through porcelain the casein is left behind and the albumin passes into the filtrate. I find, however, that the whole of the proteids are removed from solution, and have not detected albumin in the filtrate. Whether the difference in results is due to the use of porcelain of different quality I am unable to say; but it may be due to the fact that the difference in concentration of the milk and the filtrate, owing to the removal of fat and proteids, has not been taken into account, and as by neglecting this the casein would be found too low, there would be consequently a difference which might be taken as albumin.

The method of allowing for the difference of concentration is to calculate the constituents of the filtrate to the original milk by multiplying by the percentage of water in the milk, and dividing by the percentage of water in the filtrate; this I believe is correct, and is the most convenient method of making the correction.

I have also made some examinations of whey produced by the action of rennet on milk.

The following are the mean results I have obtained:

	Milk.	Serum from Fresh Milk.	Serum from Heated Milk.	Whey.	Whey Heated.
Total solids ...	12·83	5·09	5·03	6·21	6·12
Fat	4·01	—	—	—	—
Sugar	4·45	4·45	4·44	4·45	4·45
Proteids	3·46	—	—	1·24	1·16
Other solids ...	0·16	0·16	0·12		
Ash	0·75	0·48	0·47	0·52	0·51
CaO	0·17	0·054	0·045	0·051	0·047
P_2O_5	0·22	0·097	0·094	0·103	0·095
CO_2 (combined) ...	0·016	0·016	0·013	not estimated	
Total nitrogen ...	0·54			0·129	0·113
Proteid nitrogen ...	0·54			0·068	0·047
Albumose nitrogen	—			0·061	0·066

Of the total nitrogen of the milk, the casein nitrogen was 0·477 per cent., and the albumin nitrogen 0·063 per cent.

The first point to which attention must be drawn is that the alkalinity of the ash (which is expressed as CO_2) is identical in the milk and the serum; this was found to be the case in three samples. The only conclusion to be drawn from this is that the salts combined with the proteids contain equal amounts of acid and base.

By subtracting the serum lime and phosphoric anhydride from the totals we get the lime and phosphoric anhydride combined with the proteids. The following is the composition of the matter separated by porcelain:

Proteids	3·46
Casein nitrogen	0·477
Albumin nitrogen	0·063
CaO	0·116
P_2O_5	0·123
Ash	0·27

It will be convenient at this stage to deduce similarly the composition of the curd separated by rennet. This is:

Curd nitrogen	0·411
CaO	0·119
P_2O_5	0·117
Ash	0·23

While there are left in solution:

	Proteid nitrogen	0·068
and	Albumose nitrogen	0·061

Hammarsten gives the following composition for casein, the curd produced by rennet, and the proteid of the whey:

	Casein.		Curd.		Whey Proteid.	
	Found.	Calculated.	Found.	Calculated.	Found.	Calculated.
Carbon	52·96	52·96	52·88	52·95	50·33	49·72
Hydrogen	7·05	7·03	7·00	7·00	7·00	6·97
Nitrogen	15·65	15·64	15·84	15·88	13·25	13·18
Phosphorus	0·85	0·84	0·99*	0·98	—	—

From these figures I have ventured to deduce formulæ, not as absolutely correct, but probable near approximations. They are:

Casein	$C_{102}H_{258}N_{41}SPO_{52}$
Curd	$C_{140}H_{222}N_{36}SPO_{44}$
Whey proteid	$C_{22}H_{37}N_5O_{10}$

If we assume that casein separated by porcelain is

$$C_{102}H_{255}N_{41}SPO_{52}CaNa, \tfrac{1}{2}(Ca_3P_2O_8),$$

and that it is split up by rennet into

$$C_{140}H_{220}N\ SPO_{44}Ca, \tfrac{1}{2}(Ca_3P_2O_8) \text{ and } C_{22}H_{87}N_5O_{10},$$

we shall find the following figures for 0·477 casein nitrogen:

CaO	0·119
P_2O_5	0·121
Na_2O	0·026
Total ash	0·266

This would be split up by rennet into

Curd nitrogen	0·419
Albumose (whey proteid) nitrogen	0·058
Ash	0·24

---

* I have inserted this figure.

d the curd would contain the same amounts of lime and phosphoric anhydride as e casein.

As the proteid nitrogen is a little higher (0·068) in the whey than the albumin trogen (0·063), and as, moreover, traces of proteid phosphorus were found both by eumeister's method and that described in "Dairy Chemistry," p. 113 (both of which reed), it is probable that there is a little curd left in the whey, most likely in spension, as the whey is not quite clear.

The very close concordance of the figures throughout affords grounds for pposing that the view put forward above is correct. Casein may be regarded as a bstituted phosphoric acid, the substituted group having itself an acidic function; is in milk is combined with a molecular proportion of calcium phosphate. A rther confirmation of this view is obtained by adding to milk the minimum uantity of acid necessary to just curdle on boiling. If a mineral acid (sulphuric or ydrochloric acid) is used, the quantity necessary to just curdle on boiling is about ·6 c.c. of normal acid per litre of milk; the quantity calculated to replace the sodium the formula for milk containing 3·0 per cent. casein, is 8·3 c.c., and the acidity of he serum (16·8°) is practically equal to that of the original milk (16·7° after boiling).

On curdling milk with rennet, and estimating the acidity of the whey, it is und to be less acid; the mean acidity of milk was found to be 19·0 c.c. normal lkali per litre, and the whey (after correcting for the difference of volume due to emoval of curd) was 10·6 c.c. normal alkali per litre, or a difference of 8·4 c.c., a gure which is in entire agreement with the view expressed above.

The results on the serum of milk heated to 100° C., which were originally cluded to study the changes in the albumin, do not show that any great change ccurs in the salts on heating; there is a slight deficiency of lime—0·009 per cent.—hich is nearly equivalent to the deficiency in the phosphoric acid—0·003 per cent., lus the deficiency in the carbonic acid—which is derived from the combustion of e citrates of milk; this indicates that a slight deposition of calcium citrate and hosphate occurs on heating. Attempts were made to estimate the lime in the ight deposit, but, owing probably to the relatively large bulk of water necessary to ash this, only 0·0012 per cent. on the average was found. It was found also that boiling milk the acidity was reduced from 19·0 c.c. normal alkali per litre to 6·7 c.c. This is most probably due to the expulsion of free carbonic acid, and is quivalent to 0·0044 × 2·3 = 0·01 per cent., or, roughly, 50 c.c. per litre, which is bout the quantity found to exist in milk.

The amounts of lime and phosphoric anhydride in heated whey are almost xactly equal to those in the serum from heated milk, indicating that in addition a partial separation of albumin, a slight deposition of calcium salts occurs.

With regard to the acidity of milk, this can only be due to the presence of cid phosphates and citrates. I find experimentally that while tri-sodium citrate is eutral to phenolphthalein, the neutral phosphate is expressed by the composition $a_{1\cdot95}H_{1\cdot05}PO_4$ in aqueous solution and $Na_{1\cdot97}H_{1\cdot03}PO_4$ in 5 per cent. milk-sugar olution, and this appears to account for the fact that milk just neutralized to henolphthalein is slightly reddened by dilution; the end-reaction is not very sharp.

The acidity of the milk, added to the alkalinity of the ash, give a datum for the estimation of the citric acid in milk. The acidity of boiled milk is 16·8 c.c. normal alkali per litre to phenolphthalein, and as this is due to acid phosphates, this is equal to $16{\cdot}8 \times \frac{2}{0{\cdot}97} = 34{\cdot}6$ c.c. for chemical neutrality, which is equal to $\frac{34{\cdot}6 \times 0{\cdot}063}{1{\cdot}032 \times 10} = 0{\cdot}218$ per cent. citric acid; the alkalinity of the ash as CO_2 is 0·016 per cent. $= 0{\cdot}016 \times \frac{63}{22}$ $= 0{\cdot}046$ per cent. citric acid, or a total of 0·264 per cent. We may presume that, as very little calcium citrate is deposited on boiling, it exists in milk as $R_2HC_6H_5O_7$, and the citrates would account for 14·4 c.c. normal alkali per litre, and the remainder is accounted for by the phosphate RH_2PO_4; this is equivalent to 0·020 per cent. P_2O_5 being present as RH_2PO_4, while 0·077 per cent. is present as R_2HPO_4.

The item returned in the analysis as other solids agrees fairly well with the percentage of citric acid deduced, when allowance is made for the carbonic acid in the ash and the hydrogen replaced.

In conclusion, the following shows the distribution of the phosphoric acid of the milk:

P_2O_5 as casein, combined with CaNa and ...	0·0605	per cent.
P_2O_5 as $Ca_3(PO_4)_2$	0·0625	,,
P_2O_5 as R_2HPO_4	0·077	,,
P_2O_5 as RH_2PO_4	0·020	,,
Total P_2O_5	0·220	,,

and the analysis of the milk may be written:

Water	87·17	per cent.	
Fat	4·01	,,	
Sugar	4·45	,,	
Casein, with the salts in combination	3·33	,,	yielding ash 0·27 per cent.
Albumin...	0·40	,,	
Salts in solution	0·73	,,	,, ,, 0·48 ,,
Total ...	100·09	,,	

My conclusions differ from those of Söldner in that—

(i.) I consider one-third of the base with which casein is combined in milk to be soda and not lime.

(ii.) I consider that casein forms a molecular compound with calcium phosphate.

(iii.) I express the citrates as dibasic, and not as tribasic, and, consequently, our proportions of mono and dibasic phosphates do not agree.

Before finally concluding, I would point out some objections to my views. First of all, there are possible errors of analysis; and these, as the absolute quantities dealt with are small, may be relatively large. Then I have taken no account of the small amounts of magnesium which may replace calcium; I actually find a rather larger quantity of phosphoric anhydride in the $Ca_3(PO_4)_2$ than in the casein, while they should be equal; but against this there is the phosphorus of the lecithin of the fat and the higher percentage of phosphorus in Storch's mucoid proteid, which would give phosphoric anhydride, and which would be included in the phosphoric acid as calcium phosphate. Finally, I have not assumed that albumin is in combination with bases. I am inclined to disregard these objections, in face of the facts supporting my view.

DISCUSSION.

Mr. BLOUNT said that, for a purpose other than filtration, he was concerned in trying to use "biscuit" porcelain, which he hoped would prove quite impervious, not merely to liquids, but to gases. "Biscuit" porcelain, as it was known in the laboratory, was an extremely impervious material for all ordinary purposes; but it was conceivable that, if heavy pressure were used, it might suffice to filter a turbid liquid very slowly, and to effect important separations. He had himself, however, tried to filter a very turbid and refractory liquid under heavy pressure through "biscuit" porcelain, and had failed entirely to get even a drop through; and he would like to hear whether the porcelain used by Mr. Richmond was of some more porous grade than the kind generally used for laboratory vessels.

Dr. RIDEAL thought that the Society's hearty thanks were due to the author for the papers on this subject which he brought before them annually. Apart from statistical matter, they included references to interesting points bearing on the question of milk constitution—such, for instance, as the valuable suggestions which the present paper contained as to the constitution of the acids present in milk.

The PRESIDENT (Dr. Voelcker) said that in previous reports Mr. Richmond had drawn attention to the fact that each year the average percentage of fat decreased a little. In the results now given for 1900 this decrease continued to be shown, its amount being practically the same as in previous years—namely, about 0·1 per cent. It was to be noticed that the average figures for solids-not-fat, in the case of both the morning and the evening milk, were identically the same for 1900 as for 1899, and the variation in fat between morning and evening milk was also the same as in 1899, the average variation being under $\frac{1}{2}$ per cent. in each case. This was a point of considerable importance in view of what was brought out at the meetings of the Departmental Committee on milk standards, when it was sought to show that much wider differences existed between morning and evening milk than were exhibited by Mr. Richmond's figures. It should, of course, be clearly understood that the morning milk here referred to was the milk actually drawn in the morning, not the milk which was distributed in the morning, and which was probably taken overnight. He agreed with Dr. Rideal as to the value of the addenda which Mr. Richmond usually made to his annual statistics. The points brought out in the present paper with regard to the forms of combination in which lime occurred with the other constituents of the milk were not only of great interest, but of far-reaching importance. Considering the important influence which lime salts exerted in connection with the action of rennet and the formation of curd, there was indicated the necessity for still further investigation into the composition of milk, and into the manner in which the different constituents were bound together.

Mr. RICHMOND said that, as in past years, the great bulk of the work recorded in the paper had been done by Mr. J. B. P. Harrison and Mr. C. H. Rosier, his assistants, whose help he desired duly to acknowledge. It was quite true that fo practically the last twenty years there had been a small but distinct fall in fat, bu the average annual decrease was considerably less than 0·1 per cent.; and in 1901 as far as could be told at present, probably a break would occur in the continuity o the decrease, and there would be a rise in the average percentage of fat. The variation in fat between morning and evening milk had been practically the same for th last twenty years—namely, from 0·3 to 0·4 per cent., and it was only extremely rarel that any greater variation occurred. The only occasions on which he had notice large variations were when there was a large difference in the length of the interval between milking-times. If, for instance, the intervals were eight hours and sixtee hours respectively, the milk after the sixteen-hour interval would be found sometime to contain as little as 2½ per cent. of fat, while the milk after the eight-hour interva might contain as much as 5 or 6 per cent. But the intervals were usually very muc more even. He had used two kinds of porcelain, one being that used in the Pasteu filter, and the other that sold under the name of "porous balloon filters." Probabl both of these would be much more porous than the kind mentioned by Mr. Blount.

Mr. Blount observed that it would seem desirable that some definite standar of porosity should be adopted.

Mr. Richmond said that he had found that with both of the two kinds of poro lain he had used—which certainly differed from one another in porosity—the protei were completely removed from the milk; and this being so, he had not investigat the matter further.

THE RELATION BETWEEN SPECIFIC GRAVITY, FAT, AND SOLID NOT-FAT IN MILK.

By Norman Leonard, B.Sc.

In a former paper (Analyst, xxv., 67-69) the application of the method of lea squares to the results of analysis of 137 samples of milk was described, and th most probable values of the constants s and f in the equation $G = Ss - Ff$, represen ing the relation between degrees of specific gravity (G), percentage of fat (F), ar of solids-not-fat (S), were found by that method to be 3·905 and 0·815 respectivel From these results was deduced the equation $F = 0{\cdot}827T - 0{\cdot}212G$ for calculating t fat from the total solids (T) and the specific gravity. This formula, like most of t milk formulæ which have been proposed, is based on the assumption that the speci gravity of milk is raised or lowered by a constant amount for each per cent. *by weig* of solids-not-fat or fat respectively. Reasoning by analogy from the properties other solutions, it would seem probable, however, that the analytical results mig be better represented by a formula in which the influence on the specific grav was assumed to be proportional to the percentage *by volume* of the constituents, otherwise expressed, in which the percentages by weight exercised a const influence on the specific volume, instead of the specific gravity. Such a formul $G/D = S\sigma - F\phi$, where D is the density of the milk (water = 1)—was constructed

Richmond (ANALYST, xiv., 121-131), who found, however, that no superior accuracy was obtained by its use.

I have now calculated the value of the constants σ and ϕ by the method previously employed, and have obtained the formula $G/D = 3{\cdot}775S - 0{\cdot}743F$, whence $F = 0{\cdot}836T - 0{\cdot}221G/D$. The results given by this formula are scarcely so accurate as those given by the simpler formula, as will be seen from the following comparative table:

	Formula. $F = 0{\cdot}827T - 0{\cdot}212G.$	Formula. $F = 0{\cdot}836T - 0{\cdot}221G/D.$
Average error	−0·01	+0·01
Probable error	±0·078	±0·081
Percentage of errors not exceeding 0·1	70	67
Percentage of errors not exceeding 0·2	90	89

The fats calculated by the new formula are, on the average, about 0·02 per cent. higher than those given by the old formula. The difference is greatest in the case of milks with high solids and high specific gravities, whilst with milks having low solids and low specific gravities the two formulæ give more concordant results. Hence the new formula rather accentuates the error inherent in the old formula of giving too high fats when the solids-not-fat are high (*cf.* ANALYST, xxv., 68; xvii., 169-171).

The seasonal variations in the accuracy of the calculated fats are not appreciably different for the two formulæ. The specific gravities in solution of the solids-not-fat and fat, as calculated from the values found for σ and ϕ, are 1·606 and 0·931 respectively, results which compare favourably with those—1·613 and 0·939—calculated by Richmond (ANALYST, xiv., 121-131).

NOTE ON SUBLIMED SULPHUR.

By NORMAN LEONARD, B.Sc.

THE "sublimed sulphur" of the British Pharmacopœia "should not have any action upon litmus," but according to my experience it is rare to meet with specimens absolutely devoid of acidity. Those which I have examined were found to contain from 0·02 to 0·25 per cent. of sulphuric acid, as determined by titrating the aqueous extract of the samples; the average of eight samples was 0·08 per cent. Part of this acidity may be due to oxidation in the process of sublimation, but it may also be formed by exposure to air and moisture. Thus, a quantity of sublimed sulphur which had been rendered perfectly neutral by repeated washing with water and kept in a stoppered bottle whilst still moist was found to be very faintly acid after two weeks, and decidedly acid after three months, whilst at the end of four years the sample contained 0·2 per cent. of sulphuric acid. The latter was determined both by titration and by precipitation as barium sulphate; no sulphurous acid could be detected. A specimen of sulphur which had been thoroughly washed and subsequently dried at 100° showed, after keeping for four years, a barely perceptible acid reaction, corresponding with the presence of not more than 0·0025 per cent. of sulphuric acid.

I am not aware that the slow oxidation of sulphur in this way has been previously noticed.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

Lactic Acid in Wine. **R. Kunz.** (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1901, iv., 673-683.)—The author states that optically inactive lactic acid is a normal constituent of all wines, often in considerable quantity. The following method is recommended for the determination of this acid in wine: 200 c.c. of the wine are rendered alkaline by the addition of barium hydroxide, and are then evaporated in a porcelain basin to about two-thirds of the original volume. After cooling, the whole is made up again to 200 c.c., well shaken, and filtered; 150 c.c. of the filtrate are now evaporated to a thin syrupy consistency, at the same time passing a current of carbon dioxide through the liquid. When cold, dilute sulphuric acid is added in excess, and the whole liquid and precipitate, after dilution with water, extracted with ether in an apparatus similar in principle to that described in the ANALYST, 1892, xvii., 44. The extraction is complete in eighteen hours. Thirty c.c. of water are added to the ether extract, and the ether is driven off on the water-bath. The aqueous solution of the extract is then distilled in a current of steam until all volatile acids have been removed, which is attained when the distillate amounts to 600 to 800 c.c. The residue is made alkaline with barium hydroxide, heated for fifteen minutes on the water-bath, when it should still remain alkaline, and evaporated to about 10 c.c., meanwhile passing a stream of carbon dioxide through the liquid. This residue is then taken up with 40 c.c. of water, made up to 150 c.c. with 95 per cent. alcohol, shaken, and filtered. One hundred c.c. of the filtrate are evaporated until the alcohol is driven off, then made acid with hydrochloric acid, precipitated with sodium sulphate, and the weight of barium sulphate obtained calculated into lactic acid. The results of a number of determinations of lactic acid in wines are given, the quantities found being from 0·1185 to 0·7340 gramme per 100 c.c. W. P. S.

The Detection of Cocoanut Oil in Butter and Margarine. **L. Vandam.** (*Ann. de Pharm. de Louvain*, 1901, 201; through *Journ. Pharm. Chim.*, 1901, vi., 273-276.)

1. *Determination of Fatty Acids Soluble in Alcohol.*—Five grammes of the fat are saponified with 25 c.c. of an alcoholic solution of potassium hydroxide (8 grammes per litre of alcohol of specific gravity 0·832). An additional amount of the same

alcohol is then added so as to form, with the initial 25 c.c., alcohol of the required percentage after dilution to 100 c.c.

The soap solution is now diluted to 100 c.c. with water, cooled to 15° C., and again made up to the mark. It is then decomposed with 25 c.c. of standard sulphuric acid equivalent to the potassium hydroxide solution in strength, and the liquid cooled very slowly to 15° C., kept at that temperature for some time, then filtered, and the soluble fatty acids in the filtrate titrated with $\frac{N}{2}$ sodium hydroxide solution. The fatty acids on the filter are dried, and their volume determined when suspended in 10 c.c. of the alcohol. The volume of the precipitated sulphate is also determined and taken into account.

In this way the author obtained the following results, representing the fatty acids soluble in different strengths of alcohol at 15° C., expressed in terms of $\frac{N}{2}$ sodium hydroxide solution :

	ALCOHOL.					
	50 per cent.	55 per cent.	60 per cent.	65 per cent.	70 per cent.	80 per cent.
Cocoanut Oil	4·3	13·3	44·2	44·0	—	—
Butter	—	8·5	10·9	25·8	27·7	30·0
Margarine	—	—	3·6	9·5	22·8	26·2

Five other samples of butter of known purity were also tested with 60 per cent. alcohol, and gave results ranging from 10·3 to 11·8.

2. *Determination of Fatty Acids Soluble in Alcohol but Insoluble in Water.*—Fifty c.c. of the solution in 60 per cent. alcohol obtained in the preceding test ar evaporated to three-fourths of the volume, then mixed with boiling water, cooled at the ordinary temperature, and the supernatant liquid filtered. The precipitated fatty acids are washed twice with boiling water, cooled, and filtered. Finally, they are dissolved in boiling alcohol and titrated with $\frac{N}{2}$ sodium hydroxide solution.

The following results were thus obtained : Butter (4 samples), 4·6 to 5·2 ; margarine, 3·1 ; and cocoanut oil, 42·0. C. A. M.

A New Test for Saccharin. **A. Leys.** (*Ann. de Chim. anal.*, 1901, vi., 201-206.)—If 10 c.c. of a very dilute solution of copper sulphate or of a ferric salt be mixed with two or three drops of hydrogen peroxide, and warmed with an aqueous solution of an unsaturated organic compound, an intense brown coloration is produced. This reaction is obtained with salicylic acid, benzoic acid, cinnamic acid, saccharin, acetanilide, ortho-phenylene-diamine, antipyrine, pyrrhol, thiophen, and furfural. No colour is obtained with alcohol, acetic acid, lactic acid, sucrose, allyl acetate, fumaric acid, menthol, or turpentine.

The brown coloration given by saccharin can be prevented by the addition of a trace of sulphuric acid, and even after warming the mixture for a few moments only a faint yellow tint is produced. After a short time two drops of a very dilute solution of ferric chloride are added, and a violet coloration is obtained. This reaction, which is due to formation of oxybenzoic acid, is also given by benzoic acid. By using very dilute solutions of ferric chloride and hydrogen peroxide in the cold, the addition of the acid is rendered unnecessary.

In testing milk for saccharin, 50 c.c. are treated with 100 c.c. of a 10 per cent. solution of potassium bisulphate, containing 10 c.c. of 99 per cent. alcohol, and the precipitated casein filtered off. The filtrate is shaken with two portions of ether, emulsification being prevented by the addition of 2 drops of alcohol, the ethereal extracts evaporated to dryness at 90° C., and the residue dissolved in 5 c.c. of boiling water. When cold, the solution is tasted, and, if sweet, is tested with 2 drops of a very dilute solution of ferric chloride, and 2 c.c. of dilute hydrogen peroxide, and left for thirty minutes. In the presence of saccharin a violet tint is slowly developed.

The ferric chloride solution is prepared by diluting 2 c.c. of a solution of 30° Bé. to 100 c.c., and the hydrogen peroxide by diluting 1 c.c. of the ordinary 10 volume solution to 200 c.c.

C. A. M.

An Improvement on the British Pharmacopœia Santonin Test. P. Pain. (*Pharm. Journ.*, 1901, lxvii., 131.)—The test consists in warming a few crystals of santonin with 2 or 3 c.c. of ethyl nitrite solution (British Pharmacopœia strength). On the addition of a few drops of potassium hydroxide solution, a fine rose-red coloration is produced. No colour is produced until the potassium hydroxide is added, and this serves to distinguish it from such bodies as aloin and resorcin, both of which give a red colour with ethyl nitrite alone. Thymol, with the same test, yields a dark yellow solution. One milligramme of santonin can be detected by this method.

W. P. S.

Determination of Cocaine. W. Garsed and J. N. Collie. (*Pharm. Journ.*, 1901, lxvii., 222 and 254.)—The method which the authors found to be the most satisfactory one consists in slowly adding an excess of $\frac{N}{10}$ iodine solution to the cocaine solution, filtering off the brown precipitate formed through an asbestos filter, and titrating back the iodine in the filtrate with $\frac{N}{10}$ sodium thiosulphate solution. The presence of ecgonine does not interfere with the results if $\frac{N}{100}$ iodine solution be used; but benzoyl ecgonine makes the results too high. As, however, this substance is not extracted by either ether or petroleum spirit from alkaline aqueous solutions, it may be readily separated from cocaine.

W. P. S.

The Determination of Aconitine in Extracts of Aconite. H. Ecalle. (*Journ. Pharm. Chim.*, 1901, xiv., 97-102.)—This method is based on the precipitation of aconitine by silico-tungstic acid. The silico-tungstate is washed, dried, and ignited, leaving a mixture of tungstic and silicic acids, $12WO_3 . SiO_2$.

According to Bertrand (ANALYST, 1899, 187), the weight of the acids, multiplied by the factor 0·907, gives the corresponding amount of aconitine, but the author has found by test experiments that this factor gives too high results.

Bertrand's factor was based upon the assumption that the silico-tungstate contained 4 molecules of the alkaloid, but the author obtained results in close agreement with theory by calculating the factor from the formula:

$$12WO_3 . SiO_2 . 2H_2O . 3\tfrac{1}{2}Alk. nH_2O ;$$

and he therefore adopted 0·793 as the coefficient.

In determining the aconitine in a tincture of aconite, a definite weight (125 grammes) is evaporated on the water-bath, and the cold residue treated with 6 to 7 c.c. of dilute nitric acid (1 : 10). The mixture is transferred to a separating funnel, rendered alkaline with 3 to 4 c.c. of ammonium hydroxide, and extracted with successive portions of 100 c.c. of ether, of specific gravity 0·720, at 15° C., until a few drops of the solution on evaporation give no reaction with Mayer's reagent.

The united extracts are shaken with 6 to 7 c.c. of nitric acid (1 : 10) to which have been added 12 to 15 c.c. of water. The acid layer is withdrawn, and the ether washed with water until the washings are no longer acid. The acid extract and washings are gently heated to expel any ether, then cooled, and the alkaloid precipitated with 7 to 8 c.c. of a 5 per cent. solution of silico-tungstic acid, in the presence of an excess (12 to 15 c.c.) of 10 per cent. nitric acid.

The liquid is heated nearly to the boiling-point, then cooled, and allowed to stand for twenty-four hours. The precipitate is collected, washed, dried, ignited in a weighed porcelain crucible, and its weight multiplied by the factor 0·793.

(Probably the author and Bertrand were working with different kinds of aconitine.—W. C.) C. A. M.

Assay of Extractum Cinchonæ Liquidum. F. H. Alcock. (*Pharm. Journ.*, 1901, lxvii., 90.)—Five grammes of the extract are weighed out into a 4-ounce white glass bottle fitted with a good cork. Fifteen c.c. of benzolated amylic alcohol and 10 c.c. of normal alcoholic potash solution are now added; the bottle is shaken and allowed to stand in a warm place for a few minutes. The *clear* liquid is then poured off into a separator, the contents of the bottle again shaken with 15 c.c. of benzolated amylic alcohol, and poured off after a few minutes. After a third extraction with benzolated amylic alcohol, the contents of the separator are washed with two or more separate quantities of 5 c.c. of water, with or without the addition of 1 c.c. of a 10 per cent. solution of ammonia, shaken well, warmed, allowed to settle, and the aqueous liquid removed. This contains all the glycerin and often a trace of alkaloid. The solvent liquid, now free from colouring matter, alkali, and glycerin, is extracted three times with acidulated water. This aqueous extract is then treated according to the official method. The slimy residue does not retain any alkaloid.

W. P. S.

The Determination of Phenol when Mixed with Resinous Substances. J. C. Thresh. (*Pharm. Journ.*, 1901, lxvii., 138.)—Having to determine the amount of phenol in samples of carbolized gauze, in which the phenol is "fixed" to the gauze by the aid of resin, the author used the following process: About 20 grammes of the gauze were placed in a flask holding about 700 c.c., and connected to a condenser. Five hundred c.c. of water acidulated with hydrochloric acid were then added, and a few fragments of granulated zinc. After slowly distilling over about 300 c.c., practically the whole of the phenol was in the distillate, and was determined by the usual bromine method. The process gave good results when carried out on test samples. W. P. S.

ORGANIC ANALYSIS.

Notes on some Vegetable Waxes. M. Greshoff and J. Sack. (*Rec. trav. chim. des Pays Bas*, 1901, xx., 65; through *Chem. Zeit. Rep.*, 1901, 177.)—The wax obtained from the leaves of the plantain (pisang) forms white crystalline masses melting at 79° to 81° C., and having the specific gravity of 0·963 to 0·970 at 15° C. The wax is only slightly soluble in boiling ethyl alcohol, but readily dissolves in hot turpentine, amyl alcohol, and carbon bisulphide. Its acid value is 2 to 3; its saponification value, 109. On hydrolysis it yields "pisang-cerylic acid," $C_{24}H_{48}O_2$, which melts at 71°. "Pisang-ceryl alcohol," $C_{13}H_{28}O$, melts at 78°. The *Ficus ceriflua* gives a wax which is brown outside, yellowish in the centre of the mass, and has a specific gravity 1·015. It is soluble in all the usual solvents at the boiling-point, and by continued boiling it may be almost entirely dissolved in ethyl alcohol, separating again when cold. After purification with alcohol the wax melts at 61° C., and yields "Fico-cerylic acid," $C_{13}H_{26}O_2$, melting at 57°, and "Fico-ceryl alcohol," $C_{13}H_{28}O$, melting at 198°. By dry distillation it gives an aqueous distillate of acetic and propionic acids, and an oily liquid. The latter contains a colourless fluorescent hydrocarbon $C_{14}H_{26}$ which boils at 220°, and has an odour like petroleum and turpentine; also two crystalline bodies: an acid, $C_{12}H_{24}O_2$, melting at 55°, and an alcohol, $C_{44}H_{88}O$, melting at 51°. On destructive distillation pisang wax yields a hydrocarbon $C_{18}H_{34}$ boiling at 280°, and an acid $C_{27}H_{54}O_2$ which melts at 58°, and is therefore not identical with cerotinic acid. Beeswax on distillation gives a hydrocarbon $C_{15}H_{30}$, boiling between 240° and 250°, identical with one of the constituents of petroleum; also a solid body (perhaps $C_7H_{14}O_2$) melting at 63°, and a second substance melting at 56°, which evidently belongs to the olefine series. F. H. L.

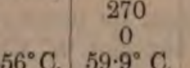
Mixed Glycerides in Natural Fats. D. Holde and M. Stange. (*Berichte*, 1901, xxxiv., 2402-2408.)—Henriques and Künne (ANALYST, xxiv., 185) confirmed Heise's statement of the occurrence of oleodistearin in Mkani fat, and their work on this subject has since been continued by the authors, who have investigated a number of specimens of olive oil, which has usually been regarded as essentially a mixture of various triglycerides.

An ethereal solution of the olive oil was chilled in a bath of alcohol and solid carbon dioxide at a temperature of −40° to −45° C., and the deposit repeatedly recrystallized first at the same temperature, then from smaller quantities of ether at −20° C., and finally freed from liquid glycerides by continued recrystallization from a mixture of alcohol and ether at the ordinary temperature.

In this way from 5 to 10 grammes of a solid glyceride resembling porcelain in appearance were obtained from 400 to 700 grammes of the oils. The filtrate when cooled to $-50°$ to $-60°$ C. yielded a crystalline deposit, which apparently consisted mainly of olein, since it melted at the ordinary temperature and had an iodine value of 82.

The solid glyceride melted at about 1° above or below 30° C., according to whether it had been previously fused or not, and in this respect behaved like Heise's oleodistearin.

It had the following physical and chemical constants: Potash value, 196·6; molecular weight 857 to 859·6; iodine value, 29·8 to 30·1; melting-point, 29° to 31·7° C.; fatty acids per cent. 95·82; specific gravity, 0·9948. These figures agree well with the theoretical values for oleodimargarin—



$$C_3H_5\left<\begin{matrix}C_{18}H_{33}O_2\\(C_{17}H_{33}O_2)_2.\end{matrix}\right.$$

The constants of the mixed fatty acids from the glyceride are given in the following table together with the theoretical values for margaric, palmitic, and oleic acids:

	Solid Fatty Acids of Glyceride.	Theory for Margaric Acid.	Theory for Palmitic Acid.	Liquid Fatty Acids of Glyceride.	Theory for Oleic Acid.
Saponification value	210·9; 211·9 (215·6)	208	219	199·1	198·9
Mean mol. weight	265·4	270	256	282·0	282
Iodine value ...	0·	0	0	90·0; 91·9	90·5
Melting-point ...	52°-53° C.; 54°-56° C. 54·6°, 57°, 59-61°C.	59·9° C.	62° C.	Liquid at ordinary temperature.	Liquid at ordinary temperature.

The ratio of solid to liquid fatty acids was as 2·2 : 1, whilst the theoretical ratio for oleodimargarin is 1·9 : 1. The liquid acids were separated by extracting the lead salts of the mixed fatty acids with ethyl sulphuric acid, and the oleic acid purified by extracting its barium salt with hot alcohol. It was concluded that the margaric acid was not a mixture of palmitic and stearic acids, because it was found impossible to obtain a fraction melting higher than 59° to 61° C., either by crystallization from petroleum spirit or by fractional precipitation with magnesium acetate. On the other hand, an artificial mixture of stearic and palmitic acid melting at 55° C. was readily separated by Heintz's method into its constituent fractions with the correct melting-points.

In an analogous experiment with bone oil the solid glycerides thus separated only had iodine values of 2·2 to 7·7, so that the authors concluded that it was improbable that mixed glycerides containing oleic acid were here present. C. A. M.

Characteristics of Sunflower Oil. **F. Jean.** (*Ann. de Chim. anal.*, 1901, vi., 166, 167.)—It is estimated that 100 kilogrammes of the seeds of the sunflower yield 15 to 20 kilogrammes of oil by cold expression. The oil first obtained is pale yellow and limpid, and has a neutral taste and odour. It is chiefly used as an edible oil in Eastern Russia. Oil of the second expression is somewhat more coloured, and is used as a lamp oil and in the manufacture of varnish. A sample examined by the author gave the following results:

Specific gravity at 15° C.	0·925
Oleo-refractometer reading	+22°
Saponification value	192
Iodine value	124
Critical temperature of solution (Alcohol, 94 per cent.)	104° C.
Melting-point of fatty acids	22° C.
Unsaponifiable matter	0·72 per cent.
Acidity as oleic acid	3·102 ,,
Solubility in alcohol	0·60 ,,

The oil reduced Becchi's reagent, but gave no coloration in Halphen's or Baudouin's tests. With Brulle's reagent (alcoholic solution of silver nitrate) it gave an orange coloration. When tested by Livache's method it absorbed 4·4 per cent. of oxygen in five days, and when exposed by itself to the air in a thin film it dried in nine days.

C. A. M.

INORGANIC ANALYSIS.

The Precipitation of Tin from its Sulpho-Salts and its Separation from Antimony by Electrolysis. H. Ort and W. Klapproth. (*Zeits. f. angew. Chem.*, 1901, 817.)—The authors have made a thorough examination of this problem, and claim to have found a satisfactory method. The apparatus consists of a glass dish, which contains an anode of platinum gauze and a porous cell, the upper part of which is surrounded by a rubber band to prevent creeping. This contains the anode and the anode solution. Antimony and tin, in the ordinary course of chemical analysis, are obtained as sulphides. These are dissolved in the smallest possible quantity of sodium sulphide, and made up to a definite bulk in a graduated flask. In one portion of this the antimony alone is determined, in another the antimony and tin together. The presence of polysulphides makes no difference; for as the solution is only placed in the cathode compartment, reduction is brought about by the current. But if much polysulphide is present, it is better to treat first with hydrogen peroxide. Not more than 0·25 gramme of antimony or 0·50 of antimony and tin should be present. For the determination of the antimony alone 8 grammes of sodium sulphide are added to the solution, which is then made up to 80 to 96 c.c., and poured into the glass basin. The porous cell in which the anode reposes contains 5 to 6 grammes of sodium sulphide dissolved in about 50 c.c. of water. The electrolysis is carried out in the cold with a current of a density of 0·1 to 0·2 ampère per square centimetre. In seven hours 0·25 gramme of antimony is entirely precipitated.

The determination of antimony and tin is carried out in the same kind of apparatus. The solution is placed in the glass basin with sufficient ammonium sulphate to convert all the sodium sulphide present into sodium sulphate. Further, 2 grammes of ammonium sulphate and 40 grammes of Glauber salt are added, and the liquid is made up to 80 or 90 c.c. and warmed to about 70° C. In the porous cell are placed 2 grammes of ammonium sulphide and 30 grammes of Glauber salt dissolved in 50 c.c. of water. Electrolysis is carried out with an initial potential difference of 2 volts and a current density of 0·3 to 0·5 ampère, and at a temperature of 60° to 70° C. In two hours the whole of the antimony and tin are deposited, together with much sulphur. To purify the deposit the cathode is taken out of the cell and put into another containing 25 grammes of Glauber salt dissolved in 125 c.c., and electrolysis is continued for another half-hour with 0·5 to 1 ampère.

In both determinations the following rules should be observed. The porous cell should not be introduced into the glass basin containing the cathode liquid until the anode solution has soaked through the pores, otherwise the resistance will be too high. The level of the anode solutions should be kept ½ to 1 centimetre above that of the cathode solution. The best material to use for the anode is a carbon rod about 1 centimetre in diameter in the case of weak currents. For strong currents, as in removing the sulphur, platinum should be used. It is not necessary to wash the deposits without interrupting the current. The cathode is lifted and rapidly rinsed. To remove the deposits of antimony and tin from the electrode, it is placed in a beaker containing strong hydrochloric acid. As soon as the evolution of gas ceases the electrode is rinsed, and placed in a second beaker with a solution of nitric and citric acids. A. M.

Thiocyanate as an Indicator in the Reduction of Ferric Salts before Titration. A. Ebeling. (*Zeits. öffentl. Chem.*, 1901, vii., 144; through *Chem. Zeit. Rep.*, 1901, 166.)—The author suggests an addition of 1 or 2 drops of a 10 per cent. solution of potassium thiocyanate to a ferric solution before it is reduced with sulphuric acid and zinc as a preliminary to the titration with permanganate. The red colour produced by the indicator gradually diminishes in intensity as reduction proceeds, until when all the iron is in the ferrous state the liquid is colourless. After cooling it can be titrated with permanganate in the ordinary fashion.

F. H. L.

Detection and Estimation of Nitric Acid in Combination with the Alkali Metals. E. P. Perman. (*Chem. News*, lxxxiii. (2161), 193.)—The alkali-metal nitrate is heated with a sulphate of a metal not belonging to the alkali group—*e.g.*, anhydrous potash alum—whereupon fumes of nitrogen peroxide are evolved. Oxygen is also liberated, leaving the alkali sulphate with alumina and the excess of the alum. After continuing to heat at low redness for one or two minutes the residue is weighed, the loss of weight, calculated as N_2O_5, enabling the percentage of combined nitric acid to be determined. In the case of potassium nitrate, the results work out to within 0·1 to 0·2 of the theoretical percentage. In presence of a chloride, however, the formation of volatile aluminium chloride vitiates the results. C. S.

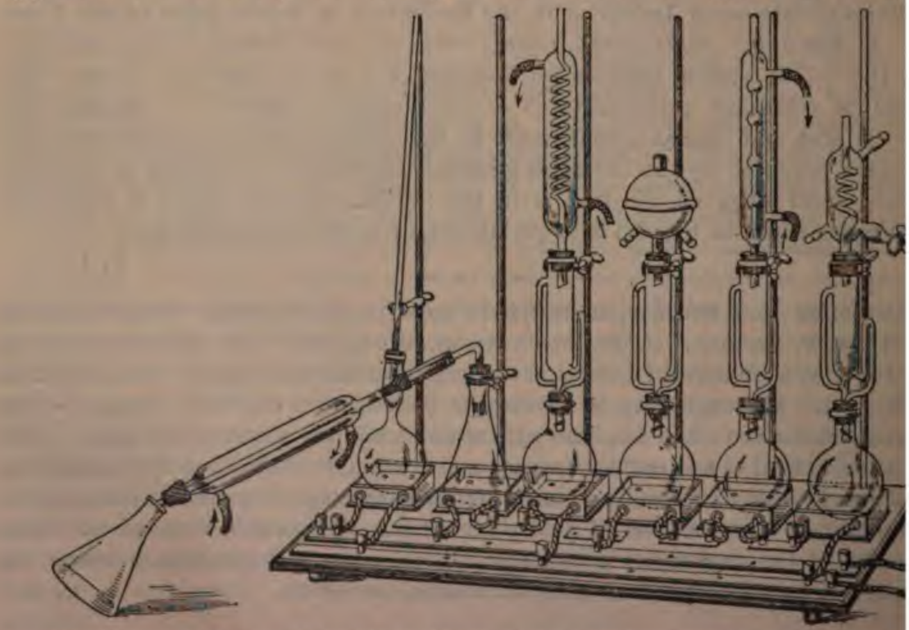
The Pulverization of Substances. W. Hempel. (*Zeits. f. angew. Chem.*, 1901, 843.)—Comparative experiments were carried out to ascertain what material is the best to use for pulverizing a hard substance such as glass. Pestles and mortars of the same dimensions, but of different materials, were used, and 10 grammes of glass were powdered fine in each. The loss of weight of the mortar and was ascertained in each case, with the following result :

Agate	0·053 gramme.
New steel	0·031 ,,
Used steel	0·005 ,,
New hard cast-iron	0·042 ,,
Used ,, ,, ...	0·014 ,,
Bottle glass	0·027 ,,

Steel gave by far the best result. Steel was also compared with porcel another experiment, and was found to be better than the latter. A steel n may be very economically made by pressing a piece of steel plate to the re shape, hardening it, and screwing it on to a wooden block. A.

APPARATUS.

An Electrically Heated Extraction Apparatus. J. Sebelien. (*Chem.* 1901, xxv., 485.)—Three advantages are claimed for this apparatus : (1) There danger of fire when volatile and inflammable solvents are being used ; (2) there steam to condense on the outside of the upright condenser ; (3) the tare flasks is not affected by deposition of "fur" from the water-bath — in fac apparatus can be safely left running with ether, etc., all night, if so desire

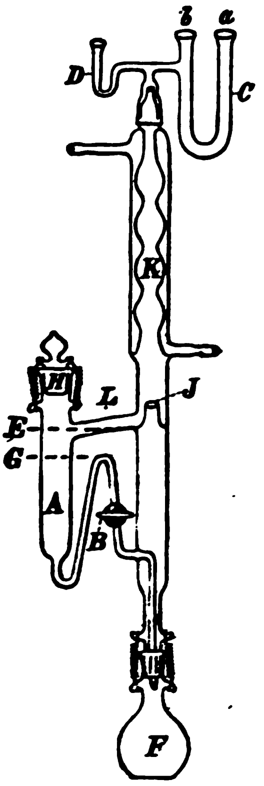


consists of a slate base, with six hot plates 10×10 centimetres in size, in whic resistances are placed ; round the plates are iron sides, to hold sand or asbes position. The connections are made with movable plugs, so that, according t temperature required, each plate can be arranged in parallel, or two or more in With a 110-volt supply, a current of 0·8 ampère will heat 100 c.c. of water t boiling-point in about twenty-five minutes, when the plate is in parallel; and, as shown in the diagram, one flask can be kept at a temperature suitable for work with alcohol, two others at a lower one to suit petroleum spirit, and the three remaining plates at a still lower heat for ether.

F. H. L.

New Fat-Extracting Apparatus. **Jerwitz.** (*Chem. Zeit.*, 1901, xxv., 437.)—This apparatus has the advantage of being made entirely of glass, the usual cork or rubber stopper above and below the extractor being done away with. The material to be extracted is placed within a paper thimble in the vessel A, which is then filled up to the level E with ether. At the close of the operation the cock B is shut, when the solvent all collects in A, and F can be removed for drying. The ground stoppers of both vessels are secured in place with spiral springs. C is a calcium chloride tube; D contains one drop of mercury to act as a safety-valve in the event of C becoming blocked.

F. H. L.



ADMIXTURE OF GLUCOSE WITH MARMALADE.

KING'S BENCH DIVISION.

(Before the LORD CHIEF JUSTICE, Mr. JUSTICE DARLING, and Mr. JUSTICE CHANNELL.)

SMITH *v.* WISDEN AND OTHERS.

(*From the "Times" of November* 23, 1901.)

THIS was an appeal by Mr. George Smith, a grocer, from the confirmation by the Horsham Quarter Sessions of a conviction under the Sale of Food and Drugs Act, 1875 (38 and 39 Vict., c. 63), section 6, whereby the appellant was convicted of selling, to the prejudice of the purchaser, a pot of marmalade not of the nature, substance, and quality demanded. The case stated was as follows: "1. The appellant is a retail grocer carrying on his business at Worthing, in the county of Sussex, and on February 7, 1901, Frederick White, Inspector under the Food and Drugs Act, 1875, in answer to his application for a 2-pound pot of marmalade, was served on behalf of the appellant with a pot of marmalade which bore a red label which, so far as is important to the case, was as follows: 'Crosse and Blackwell's pure orange marmalade, manufactured entirely from Seville oranges and warranted pure.' No indication beyond the label was given to the purchaser as to the composition of the marmalade. It was admitted before the Court of Quarter Sessions that all the formalities required by the Sale of Food and Drugs Acts were complied with by the inspector. 2. The purchase of the said pot of marmalade was made in consequence of a circular issued by the Local Government Board and received by the Chief Constable of West Sussex, of which the following is a copy: 'I am directed by the Local Government Board to inform the council that they have had under consideration the statements which have been made relative to illness in different parts of the country, alleged to be caused by the drinking of beer containing arsenic. The presence of arsenic is said to have been due to the brewing of the beer with glucose or other substitutes for sugar prepared with commercial sulphuric acid in which arsenic was accidentally present. Without expressing any opinion on this question, but with a view of pointing out a means of discovering whether any risk is likely to arise in the district from the use of beer containing injurious ingredients, the Board direct me to draw attention to the powers which the council possess under the Sale of Food and Drugs Acts of purchasing and submitting samples of beer for analysis by the Public Analyst, and to recommend that samples should be taken forthwith. I am at the same time to state that glucose and other sugar substitutes are used not only in the brewing of beer, but also in the making of jams, syrups, sweets, and similar articles of food, and the Board suggest for the consideration of the council whether it would not be well that samples of some or all of such articles which are on sale in the district should be purchased and submitted to the Public Analyst for analysis. I am to add that the Board are informed that there is no difficulty in ascertaining whether beer or other articles of food contain arsenical poison.' The conviction was in the following terms: 'In the County of Sussex Petty Sessional Division of Worthing, before the Court of Summary Jurisdiction, sitting at the Town Hall, Worthing, the 6th day of March, 1901, George Smith, of 113 and 115, Montague Street, Worthing, in the said county (hereinafter called the defendant), is this day convicted for that he, on the 7th day of February, 1901, at the parish and borough of Worthing, in the county aforesaid (by the hands of George William Smith, his son and agent) did unlawfully sell to the prejudice of one John Humphrey (the purchaser) a certain article of food—to wit, marmalade—which was adulterated with 13 per cent. of starch glucose, and was not of the nature, substance, and quality demanded by the purchaser, contrary to section 6 of the Food and Drugs Act, 1875. And it is adjudged that the defendant, for his said offence, do forfeit and pay to the clerk of this Court, Bank Buildings, Worthing, aforesaid, the sum of £1, and do also pay to Henry White, the informant, the sum of 12s. for costs forthwith. And in default of payment it is adjudged that the sums due under this adjudication be levied by distress and sale of the defendant's goods, and in default of sufficient distress that the defendant be imprisoned in his Majesty's prison at Lewes, and there kept for the space of fourteen days provided that, should the defendant pay to the governor of the said prison any sum in part satisfaction of the sum herein adjudged to be paid, including the costs and charges of the said distress and commitment and his conveyance to the said prison, the term of imprisonment shall be reduced by a number of days bearing as nearly as possible the same proportion to the total number of days for which the defendant is sentenced as the sum so paid bears to the sum for which he is so liable.—T. F. Wisden, H. Hargood, Justices of the Peace for the county aforesaid.' The certificate of the County Analyst, Otto Hehner, was as follows: 'West Sussex County Council. Sale of Food and Drugs Acts. To Superintendent W. Bridger, Worthing,—I, the undersigned public analyst for the county of West Sussex, do hereby certify that I received on the 8th day of February, 1901, from Superintendent Bridger, by registered post, a sample of marmalade for analysis (which then weighed 10 ounces), and have analysed the same, and declare the result of my analysis to be as follows: I am of opinion that the same is a sample of marmalade, or I am of opinion that the said sample contained the parts as under or the percentage of foreign ingredients as under: Starch glucose, 13 per cent. Observations.—The sample which was marked No. 231 was free from arsenic, from salicylic acid, and fruit other than orange. As witness my hand this 15th day of February, 1901, Otto Hehner, Public Analyst, at 11, Billiter Square, London, E.C.' 3. It was proved before the Court of Quarter Sessions that starch glucose is composed of 40 per cent. of dextrose, 40 per cent. of dextrine, and 20 per cent. of water. That dextrose is sugar to all intents and purposes. 4. That dextrine is a gummy substance, and has not any sweetening property whatever, and is a substance largely used for the gumming of labels and other similar purposes.

That glucose, though classed as a sugar for purposes of taxation, was not a sugar as understood by the general public. 5. It was proved that glucose had been used in the manufacture of marmalade (for a period which commenced fifteen years before the date of this case) by a large number of manufacturers, but was not so used by all, and never has been used by all. 6. That there was a general and common understanding that marmalade was a preserve composed of fruit boiled with cane or beet sugar, but that there was no legal standard for the making of marmalade, and that manufacturers varied in the recipes they used. 7. That the use of glucose to the extent contained in the analysed article was not injurious to health. That the use of glucose prevented the marmalade from crystallizing, and had a tendency to prevent mildewing and fermenting. 8. It was contended by the appellant (1) that the sale was not to the prejudice of the purchaser within the meaning of the 6th section of the Sale of Food and Drugs Act, 1875; (2) that as there is no standard for making of marmalade the addition of starch glucose was not an offence under the Act; (3) that upon the facts proved there was no evidence to sustain the conviction. 9. The Court found (1) that in asking for orange marmalade the purchaser desired to buy a substance composed of oranges cooked or preserved with cane or beet sugar, and had not consented to be served with a preserve to which starch glucose was added; (2) that the sale of the article, which contained 13 per cent. of starch glucose, was a sale to the prejudice of the purchaser; and (3) was a sale of an article not of the nature, substance, and quality of the article demanded, and affirmed the conviction and dismissed the appeal. The question for the Court is (1) Was the Court of Quarter Sessions right upon the facts proved before it in dismissing the appeal? (2) If the Court should be of opinion that the judgment of the Court of Quarter Sessions was correct, then the order of that Court dismissing the appeal with costs will stand. (3) If the Court should be of opinion that the judgment of the Court of Quarter Sessions is wrong, then this case is to be remitted to the said Court to enter judgment allowing the appeal with costs."

Mr. Bousfield, K.C., and Mr. Morton Smith appeared for the appellant; Mr. Boxall for the respondents.

The Lord Chief Justice: What evidence is there that the inspector thought he was going to get cane or beet sugar?

Mr. Justice Channell: I should think that marmalade is much older than the use of cane or beet sugar.

Mr. Bousfield: You will remember that some years ago there often used to be a layer of crystallized sugar on the top of jam. Glucose prevents that.

The Lord Chief Justice: I am not upon jam, but upon marmalade.

Mr. Bousfield, continuing, submitted that there was no evidence that there was a sale to the prejudice of the purchaser.

The Lord Chief Justice: I think you are entitled to say that if there is no adulteration something more than a different article being sold to that asked for is necessary.

Mr. Bousfield, continuing, submitted that the present case was not within the Act at all. He cited "Goulder *v.* Rook" (1901, 2 K.B., 290). The section did not apply where there was a mixture with no fixed standard, "Dickens *v.* Randerson" (1901, 1 K.B., 437), "White *v.* Bywater" (19 Q.B.D., 582). Where there was no fixed standard it could not be said that the purchaser expected to get marmalade as defined in the dictionary. The only true inference was that he expected to get that which he had been accustomed to get for years. It was found that there was nothing injurious to health.

The Lord Chief Justice: That is clear.

Mr. Boxall, for the respondents, submitted that the magistrates were entitled to rely upon their own knowledge in drawing the inference as to whether the marmalade was inferior. It was essentially a case for the magistrates to decide, "Webb *v.* Knight" (2 Q.B.D., 530). It was to the prejudice of the purchaser, Hoyle's case (4 Q.B.D., 233). The appellant had to make out he was within the exception. The magistrates were entitled to rely upon their own knowledge, "Short *v.* Robinson" (63 J.P., 295).

Mr. JUSTICE CHANNELL: I cannot help thinking that people nowadays think that glucose is something dangerous. They would not have done so two or three years ago.

The LORD CHIEF JUSTICE, in giving judgment yesterday, said that the statements in the case as affording the foundations for the findings were unsatisfactory. As to what the man asked for, the Court was concluded by the finding of the justices, but he should not have come to the same conclusion. There were many other things that were perfectly proper to put in marmalade besides sugar, and which a purchaser might not know anything about. It had been judicially decided that the difference between the article demanded and that supplied must be to the prejudice of the purchaser. The magistrates had found as a fact that there was an alternative ingredient. It was found that there was no legal standard, that there was a frequent but not exclusive use of glucose, and a variation of recipes. Glucose was found not to be injurious to health. The Act ought not to be construed as an instrument of oppression. Its object was the protection of health. In the present case an article was given to the purchaser which, if different, was rather better. There was no evidence of any inferior quality or of adulteration in the ordinary sense of the word. The conviction must be quashed.

Mr. JUSTICE DARLING and Mr. JUSTICE CHANNELL concurred.

Conviction quashed.

REPORT OF THE COMMITTEE ON FOOD PRESERVATIVES.

THE Departmental Committee on Food Preservatives appointed by Mr. Chaplin in 1899 has now issued its report. The members of the Committee were the Right Hon. Sir H. E. Maxwell, Bart., M.P., Professor T. E. Thorpe, C.B., D.Sc., F.R.S., Dr. H. T. Bulstrode, and Dr. F. W. Tunnicliffe.

The report contains a large amount of original information deeply interesting to the Public Analyst. It is a bulky volume of some 500 pages, and may be obtained from Messrs. Eyre and Spottiswoode, the price being 4s. 3d. We reprint the recommendations of the committee, and also give a summary of the opinions expressed by one dissentient member:

RECOMMENDATIONS.

"(*a*) That the use of formaldehyde or formalin, or preparations thereof, in foods or drinks, be absolutely prohibited, and that salicylic acid be not used in a greater proportion than 1 grain per pint in liquid food and 1 grain per pound in solid food. Its presence in all cases to be declared.

"(*b*) That the use of any preservative or colouring matter whatever in milk offered for sale in the United Kingdom be constituted an offence under the Sale of Food and Drugs Acts.

"(*c*) That the only preservative which it shall be lawful to use in cream be boric acid, or mixtures of boric acid and borax, and in amount not exceeding 0·25 per cent. expressed as boric acid. The amount of such preservative to be notified by a label upon the vessel.

"(*d*) That the only preservative permitted to be used in butter and margarine be boric acid, or mixtures of boric acid and borax, to be used in proportions not exceeding 0·5 per cent. expressed as boric acid.

"(*e*) That in the case of all dietetic preparations intended for the use of nvalids or infants, chemical preservatives of all kinds be prohibited.

"(*f*) That the use of copper salts in the so-called greening of preserved foods e prohibited.

"(*g*) That means be provided either by the establishment of a separate court f reference or by the imposition of more direct obligation on the Local Government oard to exercise supervision over the use of preservatives and colouring matters in ods, and to prepare schedules of such as may be considered inimical to the public ealth."

One member of the committee (Dr. Tunnicliffe), while agreeing with the report general, takes exception to one of the conclusions arrived at (paragraph 127) nd to the recommendation marked *f*. The former states that it was found to be ndesirable that copper or other poisonous metallic substance should be added to od; the latter recommends prohibition of their use. In defence of his opinion, r. Tunnicliffe urges that many articles of diet naturally contain copper, that this etal is constantly being introduced into food by the use of copper cooking utensils, nd that the copper so taken up is converted into a relatively insoluble and un- sorbable compound. He thinks that the small amounts of copper contained in reserved peas are harmless, since peas are taken in comparatively small quantities, nd that no analogy can be drawn between this compound of copper in peas and inute quantities of lead in water or arsenic in beer, both of which liquids are nsumed in large quantities. He points out that in France an order prohibit- g this addition had to be subsequently rescinded, and that in Germany, where similar prohibition exists, it is quite possible to obtain copper-greened peas in the pen market, thus showing that the enforcement of the order is difficult. Dr. Tunni- liffe points out the value of appearance in rendering foods appetizing. He thinks e public taste should not be denied the gratification of having a perennial supply f green vegetables, nor the thriving industry which produces them be destroyed. e is satisfied that excessive amounts of copper are frequently used for this purpose, ut that, after diligent inquiry, no injurious results have been traced to the use of eas containing these large quantities. He recommends that only the amount of pper absolutely necessary should be added; that such amount should be declared; nd that not more than the equivalent of half a grain of metallic copper per pound ould be added.

COMBINED APPOINTMENTS OF MEDICAL OFFICER OF HEALTH AND PUBLIC ANALYST.

HE following extract from the proceedings of the Council of the Institute of hemistry will be read with interest by all Public Analysts:

"The Council have repeatedly addressed the Local Government Board for ngland and Wales on the undesirability of the appointments of Medical Officer of ealth and Public Analyst being combined in one person. There has been a growing endency in the last few years on the part of Borough Councils and other local uthorities, generally with a view to economy, to create such combined appointments.

"On July 15 a letter on this matter was addressed to the President of the Local Government Board by the Presidents of the Institute and the Society of Public Analysts, who drew attention to the importance of requiring adequate evidence of training in chemistry from any person nominated by a local authority for appointment as Public Analyst under the Sale of Food and Drugs Acts. The opinion was expressed that evidence of a systematic course of training in general theoretical and practical chemistry, followed by special experience in the analysis of food and drugs, should be demanded of all candidates offering themselves for the position of Public Analyst. It is not suggested that it is impossible to find Medical Officers of Health who have the necessary knowledge and experience, but that the extent of the training in chemistry required to be taken by medical practitioners in the course of their professional education falls far short of that necessary for the proper conduct of the duties and practice of a Public Analyst, and that medical practitioners, as such, cannot be expected to be able to deal efficiently with such problems as a Public Analyst has to encounter. Moreover, when the appointments are proposed to be combined, the selection of candidates for the two positions must necessarily be limited.

"The nature of the duties of a medical officer may be such as to require at times his entire attention (as, for example, in the event of epidemics), the consequence being that where the appointments are combined the discharge of his duties as Public Analyst may be neglected, and the effective working of the Sale of Food and Drugs Acts necessarily obstructed.

"Apart from the question of the desirability of combining these medical and analytical appointments, it is doubtful whether such combined appointments are strictly legal. In any event, it cannot be said that they were contemplated when the Act was passed, as may be inferred from the following extract from the Sale of Food and Drugs Act, 1875:

38 *and* 39 *Vict., Chap.* 63.

"'13. Any medical officer of health, inspector of nuisances, or inspector of weights and measures, or any inspector of a market or any police constable under the direction and at the cost of the local authority appointing such officer, inspector, or constable, charged with the execution of this Act, may procure any sample of food or drugs, and if he suspect the same to have been sold to him contrary to any provision of this Act, shall submit the same to be analysed by the analyst of the district or place for which he acts, or if there be no such analyst then acting for such place, to the analyst of another place; and such analyst shall, upon receiving payment as is provided in the last section, with all convenient speed analyse the same, and give a certificate to such officer, wherein he shall specify the result of his analysis.'

"While the section above quoted clearly indicates that the Medical Officer of Health and Public Analyst should be two distinct persons, it provides an additional reason why the Medical Officer of Health of a district should not be the Public Analyst of the same district, for it states that the medical officer is one of the persons whose duty it may be to take samples for analysis, or to procure their being taken. It must be admitted that it is inadvisable that the person who procures samples, thus suspecting them to be sold contrary to the Act, should also be the person to whom they are entrusted for analysis.

"In connection with the appointment of a Medical Officer of Health, the Local Government Board have the advantage of the advice of the medical officer to the Board. But in connection with the appointment of Public Analyst—which is not a medical appointment, and can only rightly belong to the profession of analytical and consulting chemistry—there does not appear to be attached to the Board any officer who is technically conversant with the qualifications necessary for the proper conduct of the practice of an analytical and consulting chemist.

"In reply to the Council's representations, a letter was received from the Board stating that, although they have not encouraged the holding of the two offices of Medical Officer of Health and Public Analyst by the same person, they were not prepared to lay down a general rule that the two offices should not be held together. Although the Board in their reply have not dealt with all of the points raised, the Council has reason to believe that their action has been beneficial."

REVIEWS.

Select Methods in Food Analysis. By Henry Leffmann, A.M., M.D., and William Beam, A.M., M.D. Published by P. Blakiston, Son, and Co., 1,012, Walnut Street, Philadelphia. Price $2.50.

In the preface to this work it is pointed out that the book "is intended to be a concise summary of analytic methods adapted to the needs of both practising analysts and advanced students in applied chemistry." The work certainly justifies such a description. Considerable pains have been taken, particularly in avoiding unnecessary detail, to prominently bring forward such points as are essential, and in describing such principles as are involved in general commercial food analysis.

The opening chapters of the book deal with physical data, and subsequently with chemical data. On carefully reading the first twenty-six pages, into which is compressed the physical portion, one feels somewhat disappointed that it does not occupy a larger number of pages—not because the descriptions and methods are exactly curtailed, but because the information is so rarely given in such a concise form.

The second portion of the book is devoted to applied analysis. Interesting information is given on poisonous metals, colours, and preservatives, and interest attaches to the information with regard to preservatives, because the practices maintaining in this country for preserving articles of food are not necessarily the same as those in America. The articles on starch, sugar, fats, and oils are all indicative of the pains which have been taken to introduce, as far as possible, all the essential data which one requires without the unnecessary information which one so frequently finds in books of reference.

The remainder of the work covers the important ground of milk and milk products, non-alcoholic beverages, condiments and spices, alcoholic beverages and flesh foods. Fifty-eight pages are devoted to milk and milk products, and the information therein contained is thoroughly up-to-date.

Altogether the work gives one the impression of being somewhat condensed, but entirely satisfactory, and will certainly form a valuable addition to the shelves of the analyst.

We should have wished to see mention made of the Marsh test for arsenic which for so long, in some modification or other, has held its own against all othe methods of detection.

A small number of mistakes occur which have obviously crept in throug oversight. For example, on p. 23 we find Fig. 12 described as "Fig. 23"; on p. 3 the word "grains" is rather awkwardly substituted for "grammes"; and on p. 1 a more serious error occurs, where the specific gravity of butter-fat is given 0·909 to 0·914, and that of cocoanut olein as 0·907 at 100°. This obviously refers 100° F., although the corresponding figures given for other fats are obviously intend for 100° C. The type of the book is excellent, and the illustrations equal satisfactory. W. C.

THE CHEMICAL ESSAYS OF CHARLES WILLIAM SCHEELE. Translated by THOM BEDDOES. (London: Scott, Greenwood, and Co.) Price 5s. net.

The bulk of this book consists of a new edition of the original translation Scheele's twenty-five Essays by Beddoes, which appeared in 1786, and to these now added a short biography of the great chemist by J. Geddes M'Intosh. Th essays are in reality the notes of the experiments made by Scheele during his all t brief, but brilliant career. Couched in simple and terse language, they bear up them the impress of a highly conscientious mind humbly seeking the truth, an adding fact to fact until a safe deduction could be made. When one bears in min the difficulties Scheele worked under—for his circumstances were never at any tim luxurious—the primitive apparatus at his command, and the enormous amount excellent work he performed, one cannot fail to be struck with admiration at th indomitable perseverance of the man against all obstacles. The book is one whic will be read with satisfaction by all true students of science. W. J. S.

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WE are informed that the next examinations will be held at the Institute i January, 1902. The intermediate examination will occupy at least four days, com mencing on Tuesday, January 7. Examinations in the following branches of th final examination will extend over at least four days, and may commence on eithe January 7 or 14: (*a*) Mineral Chemistry; (*b*) Metallurgical Chemistry; (*c*) Physic Chemistry; (*d*) Organic Chemistry; and (*e*) The Analysis of Foods and Drugs an of Water, including an Examination in Therapeutics, Pharmacology, and Microscopy An Examination in Therapeutics, Pharmacology, and Microscopy (open to Fellow and Associates) will also be held in January next.

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VOLUME XXVII.

1902.

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The Analyst,

THE ORGAN OF THE

Society of Public Analysts.

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THE ANALYST.

JANUARY, 1902.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE monthly meeting of the Society was held on Wednesday evening, December 11, in the Chemical Society's Rooms, Burlington House. The President (Dr. J. AUGUSTUS VOELCKER, M.A., B.Sc.) occupied the chair.

The minutes of the previous meeting were read and confirmed.

Certificates of proposal for election to membership in favour of Messrs. Horatio Ballantyne, F.I.C., M. Hunter, M.A. (Oxon.), Ernest Alfred Lewis, Frederick J. Lloyd, F.I.C., Albert E. Parkes, F.I.C., and Thomas Tickle, B.Sc. (Lond.), were read for the second time; and a certificate in favour of Dr. L. T. Thorne, F.I.C., Technical Chemist, 8, Dynevor Road, Richmond, Surrey, was read for the first time.

Messrs. L. Kidgell Boseley and Martin Priest were appointed to act as auditors of the Society's accounts for the year 1901.

The following papers were read: "A Contribution to a Knowledge of the Chemistry of Cider," by Alfred H. Allen; "On the Mannitic Fermentation of Wine," by Philip Schidrowitz, Ph.D.; "On Cocoa Extracts," by E. G. Clayton; and a "Note on Commercial Zinc White and other Pigments," by E. G. Clayton.

THE DETERMINATION OF CARBON IN STEEL BY DIRECT COMBUSTION.

BY BERTRAM BLOUNT, F.I.C.

(*Read at the Meeting, November* 6, 1901.)

IN the ANALYST for June, 1900, I have discussed the question of devising a practicable method for the determination of carbon in steel by direct combustion—*i.e.*, without having recourse to the usual process of previously removing the iron by solution in potassium cupric chloride or the like solvent. I would lay emphasis on the adjective "practicable." There are many prescriptions for burning steel and collecting the carbon dioxide representing its carbon, but they hardly satisfy the condition of being workable. Either the metal has to be finely divided by mechanical means or the iron must be removed by volatilization in a current of chlorine or bromine without attacking the carbon, which is only possible if air and moisture be completely excluded—a condition difficult of attainment. Such operations fall far short in simplicity and accuracy of a method which shall be capable of oxidizing steel directly and completely in substantial fragments, such as drillings or turnings, and of yielding the whole of its carbon as carbon dioxide free from other gases absorbable by potash.

In the paper already referred to, and in another on the determination of oxygen in commercial copper (ANALYST, xxi., 57), I have pointed out that it is unnecessary for the chemist in performing analytical operations of this class to restrict himself to the use of temperatures commonly attained in the combustion furnace. With proper precautions a temperature of 1,100° C. is as manageable as is one of 800° C., and reactions which are slow and imperfect at the latter take place smoothly and completely at the former. Such difficulties as have to be overcome arise from the fact that ordinary apparatus and appliances are not as a rule designed for the production and utilization of these higher temperatures, and certain modifications in instruments and procedure are called for.

In my previous experiments I endeavoured to burn steel turnings in oxygen without admixture of any oxidizing or fluxing agent. I found that there was a considerable tendency to leave a core of unburnt metal which was usually free from carbon, but, if occurring, raised a doubt whether the combustion was complete; if heating was continued until this core was oxidized, the operation was unduly prolonged, and errors inseparable from a lengthy combustion crept in. In spite of these obstacles, the results obtained were sufficiently encouraging to warrant further study of the subject, and I turned my attention to the use of some suitable oxidant. Of all the oxidizing materials which suggested themselves, lead chromate appeared preferable, because, unlike lead oxide (no doubt an ideal carrier and flux for the purpose in hand), it does not attack porcelain violently, and, further, when temporarily reduced, as it may be by the steel even in an atmosphere of oxygen, it does not yield fluid masses of metallic lead, and consequently does not imperil the integrity of platinum. Therefore two standard refractory materials become available.

Using lead chromate, the process resolved itself into this: A weighed quantity of the steel, usually 5 grammes, in the form of drillings or turnings, often of relatively stout section, was mixed with 15 to 20 grammes of lead chromate which had been recently fused and thus freed from oxidizable impurities. The mixture was put into a large porcelain boat, the boat was placed in a long, deep tray of platinum sheet, and the whole slid into a porcelain tube. The object of the platinum tray was to prevent any chromate which might creep from coming in contact with the porcelain tube. It was found impracticable to use a platinum boat, as the hard fused mass of chromate and oxide of iron left after combustion was difficult to remove without injuring the boat. The front part of the porcelain tube contained copper oxide, and was heated in a Fletcher combustion furnace. The back part was similarly heated, but the precise spot where the boat lay was raised to a temperature considerably higher than could be reached by the combustion furnace. After many trials of different burners, a benzoline blast-lamp was found to be fairly effective for this purpose. I may remark in passing that there is an opening for a burner as handy as a common bunsen, but giving the temperatures usually requiring a blast for their production—*i.e.*, ranging from the melting-point of copper to that of cast iron. I have not yet been able to procure a burner of this description from chemical dealers, and in many operations am compelled to have recourse to a mechanical blower, when a self-contained blast-

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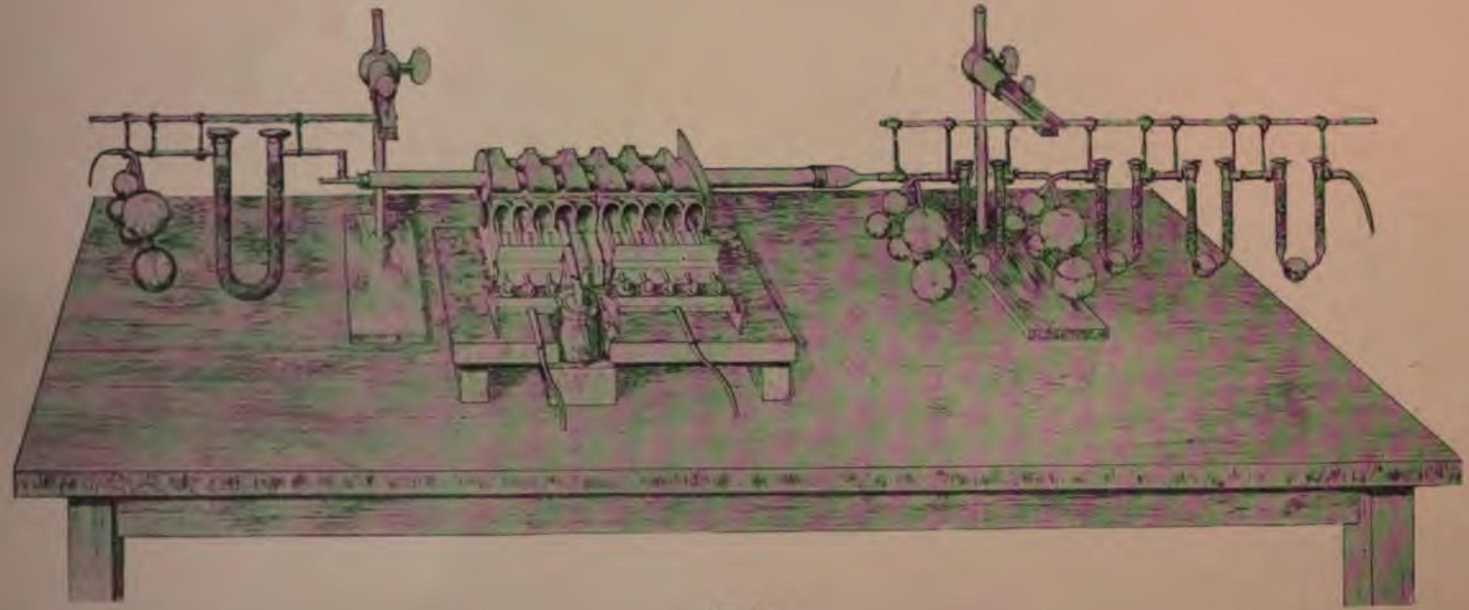

Fig. 1.

lamp of ample power would be preferable in every way. An electrical method of heating may be the best solution, but there is nothing of the kind in common use except little muffles for dental work, and I have not had time to design such an apparatus for my special needs.

The arrangement of the whole apparatus is shown in the accompanying diagram (Fig. 1).

Starting on the left, we have a potash bulb and a U-tube of soda-lime to purify the oxygen, and afterwards the air used for sweeping out the tube. The glass T-piece with free flattened end allows the boat to be seen in the tube during combustion. A Fletcher combustion furnace is used, supplemented by a benzoline blast-lamp at the point where the boat is placed. Connection at the far end of the porcelain tube is made by a glass cap on a rubber ring instead of the usual cork. This cap is drawn down so as to fit small tubing for connection with the absorbing train. The first potash bulb is empty, and serves merely for cooling. It can, if necessary, be immersed in a beaker of water. A U-tube of sulphuric acid on pumice dries the gas, and a potash bulb and similar U-tube constitute the weighed absorption apparatus. A second weighed sulphuric acid U-tube serves as a check on the efficiency of the first. It is weighed every three or four combustions, and should gain only fractions of a milligramme. A guard tube of sulphuric acid on pumice completes the train, and to this the pump can be attached.

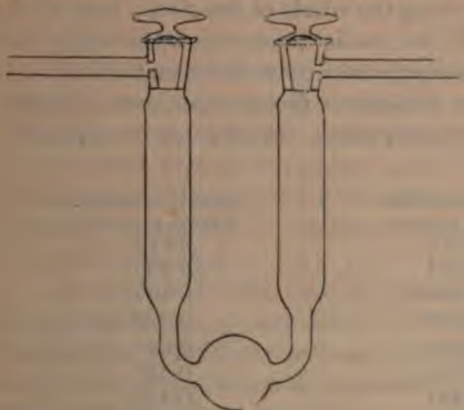
FIG. 2.

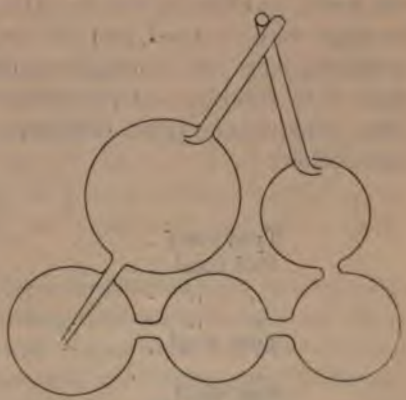
FIG. 3.

The U-tubes are designed so that the gas is compelled, not merely to traverse two columns of pumice, but to bubble through the acid. The bottom bulb forms a useful store of acid with which the pumice can be refreshed, for it often happens that when sulphuric acid on pumice alone is used, the acid on the surface of each piece may be diluted and exhausted, although there is still plenty of strong acid in the centre of each piece of pumice. The tube will then cease to act efficiently, but if it contains a store of acid actually fluid, this can be washed over the pumice in each limb, and replace the used-up acid. A diagram of the tube is shown (Fig. 2).

The potash bulb designed by my chief assistant, Mr. S. Dickson, is also novel, and an improvement on the ordinary forms. A diagrammatic sketch is given

(Fig. 3). The potash bulb differs from most apparatus of the kind by being asymmetrical. The entrance limb is larger and heavier, and the bulb hangs naturally in its proper position for absorption, so that no tying up or tilting is necessary. An extra passage through the liquid is secured by an internal tube in the first bulb, and both entrance and exit tubes project a little way into their respective bulbs, and are turned aside from the centre, their ends being also contracted. Under reasonable conditions of use splashing is almost impossible. I use these bulbs for all absorption work, and find them preferable to any other design with which I am acquainted.

When the method was carried out in this manner, satisfactory results were obtained. The high temperature employed made it necessary to insure that the drying tubes were kept in excellent condition, and were not run too long without renewal.* It was also requisite to have a good length of tube projecting beyond the furnace, to avoid over-heating the rubber connections. A porcelain tube 26 inches by 1⅛ inches was found convenient. With an adequate temperature and time of heating (about one hour), no difficulty was experienced in burning the whole of the steel; even thick turnings were oxidized, and left no core. In the earlier experiments, in which the conditions were less thoroughly understood, discrepancies occur between the standard method by solution and combustion and the method of direct combustion. In the later experiments these differences tend to disappear. The figures obtained are given below:

	Solution Method. Carbon per cent.		Direct Combustion. Carbon per cent.
Tyre steel	0·675		0·619
Rail steel	0·404		0·393
,, ,, ...	0·399		0·407
,, ,, ...	0·378		0·330
Tyre steel	0·643		0·645
,, ,, ...	0·618		0·557
Rail steel	0·401		0·419
,, ,, ...	0·432		0·435
,, ,, ...	0·430		0·408
,, ,, ...	0·457		(1) 0·422 (2) 0·393 (3) 0·438
Rail steel	0·470		0·429
Nickel steel	0·323		(1) 0·324 (2) 0·324
Tyre steel	0·459	Improved apparatus for cooling and absorption	0·445
Structural steel (plate) ...	0·288		0·277
Rail steel	0·455		0·467
,, ,, ...	0·568		0·575

It is evident from these results that the method is reliable when properly handled. But it still has several defects, due chiefly to the imperfectly refractory nature of the materials at one's command. At the temperature necessary the glaze of royal Berlin porcelain is slightly sticky, and the platinum tray adheres thereto.

---

* The efficiency of the train of absorption tubes finally adopted was tested by numerous blank experiments, and by the use of a second drying tube after the potash bulb weighed independently of the drying tube proper.

It is difficult to detach, and when forcibly removed is itself often deformed and injured, and removes fragments of the glaze, frequently leading to the breakage of the tube. The porcelain boat also adheres to the platinum tray, and sometimes can hardly be withdrawn from the tube without tearing the platinum, which is itself stuck to the porcelain tube. Various devices suggest themselves to remedy these inconveniences, and these I propose to examine when work allows. In the meantime, I am of opinion that the method will suffice to check the results obtained by the usual process in the event of a doubt arising. So much has the customary process been elaborated that there are schools of analysts who quarrel about the position of a potash bulb, and seek to standardize every piece of apparatus. All this is, of course, mischievous and absurd, and it is well to remember that the real object in view is to determine accurately the percentage of carbon irrespective of any particular procedure. Hence a process which has not yet suffered from pseudo-standardization presents some advantages.

DISCUSSION.

Mr. RAYMOND ROSS inquired whether it would be possible to use a piece of glass tube for each experiment instead of a permanent porcelain tube with boat and platinum shield. He had himself obtained very satisfactory results in carrying out with certain organic substances some combustion experiments, in which he had been obliged to use a very short furnace, by employing lead chromate alone as an oxidizing agent and conducting the combustion in a piece of glass tube about 10 or 12 inches long.

Mr. ARCHBUTT said that Mr. Blount was to be congratulated upon having, after long perseverance, at last found a method of burning steel perfectly without going through the roundabout process of decomposing with copper salts, and then burning and weighing the carbon so separated. Of course, it was to be hoped that Mr. Blount would be able ultimately so to improve the process that it would be sufficiently rapid to replace the ordinary more cumbrous method. At present it would hardly do that, for in the ordinary process a number of samples of steel would be dissolved at the same time in the double chloride of copper and potassium, and the carbonaceous residues filtered off one after another; and, as the combustion took place fairly quickly and at a comparatively low temperature, a considerable number of determinations could be carried out in one apparatus in a single day. The new method, however, as he had remarked on a previous occasion, was valuable in its present stage because it afforded a complete check upon the results of the ordinary process. Mr. Blount, in referring to the use of lead chromate, had used the text-book expression "recently fused." Surely, however, it was immaterial whether the fusion had taken place recently or not, provided that the lead chromate really had been fused and had been so kept that it could not absorb moisture or carbonaceous matter. In the course of some experiments in standardizing a pyrometer for obtaining the cooling curves of metals, he had found it possible with a small furnace, by using compressed air at about 70 pounds per square inch and coal gas under a pressure of 6 or 7 inches of water, to obtain temperatures very much higher than with gas at ordinary pressure and air supplied from a blower. He had easily melted copper in this way. Another suggestion was that a certain proportion of oxygen might be mixed with the air from the blower, by which a higher temperature would be obtained without increase of pressure. He would like to hear at what rate the oxygen was passed over the burning steel, because, from what was said about the high temperature of the gas that had to be dried, it would seem that the oxygen must be passed through at a very rapid rate. At a moderate rate—say from four to six bubbles per second—the gas might be expected to become sufficiently cool by radiation very soon after escaping from the combustion tube. If the rate were more rapid, possibly soda-lime might be a better absorbent than caustic potash for the carbonic acid. He had at one time experienced some difficulty in the absorption of the carbonic acid by potash and the weighing of the potash bulbs. His custom was not to aspirate air through the combustion furnace after burning the carbon, but first of all to pass oxygen through the apparatus and weigh the bulbs full of oxygen at intervals of ten minutes until constant, then burning the carbon and weighing the bulbs again full of oxygen at similar intervals till constant. He had found, however, that the bulbs showed a tendency to lose a few tenths of a milligramme continuously, which was ultimately traced to the not quite sufficiently hygroscopic character of the drying tube at the end. In that tube had been used, not calcium chloride—a very imperfect drying agent—but broken-up pieces of ordinary stick-potash, which was very commonly used for the purpose. Dittmar, however, in his researches on the combining proportions of oxygen and hydrogen, had demonstrated the remarkable drying powers of potash, which had been freshly fused until it contained no water. So-called pure potash, when heated in a platinum or a nickel dish, lost from 15 to 20 per cent. of water, and became perfectly anhydrous; and if this, after cooling, were broken into small pieces and used for filling the small tube at the end, no loss occurred in the potash bulbs within ten minutes or a quarter of an hour, and they could be got almost absolutely constant. He thought that if Mr. Blount were to give up aspirating air through the apparatus, and weigh the bulbs full of oxygen instead, the results would be obtained perhaps a little more quickly.

Dr. Dyer inquired whether the use of sulphuric acid for drying purposes was wholly desirable in view of the fact, which had been pointed out by Mr. Allen in the course of the discussion on Mr. Blount's previous paper on the same subject, that sulphuric acid was liable to absorb small quantities of carbonic acid.

Mr. Blount said he was afraid it would not be possible to use a glass tube as suggested by Mr. Ross, because the essence of the process was the high temperature employed. The temperature necessary was so considerable that the glass would blow out with the ordinary pressure needed to drive the oxygen through the train of apparatus. Even when chromate of lead was used, the temperature requisite in order completely to burn steel which was in stout fragments was such that the use of glass, however hard, was impracticable. He acknowledged freely that at present he had no intention of putting this method forward as capable of replacing the ordinary process. He was alive to the necessity of speed, and the method might not compete in effective speed with the older process; it was true that its real speed was greater, because, although the combustion took longer, there was no necessity for dissolving and filtering. He thought he might claim for it that it could be employed instead of the old process in any case of difficulty or doubt as to the purity of the reagents used, or when it was desired to fall back upon a totally different method, which should not involve any single step at all comparable with those of the ordinary process. He agreed with Mr. Archbutt's comment as to the term "recently fused," and, in fact, had erased it once, but, the search for a more suitable expression having been unsuccessful, he had replaced it. Clearly it was merely essential that the lead chromate should be kept clean after fusion, though it was perhaps better practice that the fusion should be recent, so as to minimize any chance of dirt being present, and that was the only excuse for the term. He had tried the use of what was known as "press gas," which, at a pressure of a few inches, was no doubt comparable with the pressures mentioned by Mr. Archbutt; then moderate pressure was hardly enough, but if gas under a pressure of a few pounds were available, it would probably not be necessary to use a benzoline blast-lamp. There was no difficulty in absorbing the carbonic acid when the oxygen was passed through at any reasonable rate. The trouble arose in absorbing the water. The use of fused potash might well be of assistance in this connection.

Mr. ARCHBUTT inquired where all the water came from.

Mr. BLOUNT said that a tangible quantity would come from the tube and apparatus, which could never be got completely dry, and in any case frequently had to stand overnight. Then, again, the copper oxide was somewhat hygroscopic, and there were a number of other sources of water which had to be reckoned with. He had never had occasion to try whether carbonic acid was absorbed by sulphuric acid, but that matter did not really bear upon the present case, because, although there might be some absorption if concentrated carbonic acid were passed over sulphuric acid, there would be none in the case of a gas containing but a small percentage of carbonic acid; one had to consider the partial pressure of the gas. These attenuated gases could be passed through even water without any appreciable loss of carbonic acid.

ARSENIC COMMITTEE.

The joint committee of the Society of Chemical Industry and of the Society of Public Analysts has now completed its investigations, and the report will be presented to the respective Societies in January.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

A Modification of the Sulphuric Acid Test for Formaldehyde in Milk. A. Gustav Luebert. (*Journ. Amer. Chem. Soc.*, xxiii., 682.)—The test is made as follows: Five grammes of coarsely-powdered potassium sulphate are placed in a 100 c.c. flask, 5 c.c. of the milk distributed over it by means of a pipette, and then 10 c.c. of concentrated sulphuric acid carefully poured down the side of the flask. The whole is allowed to stand, and if formaldehyde is present the crystals of potassium sulphate will acquire a violet colour in a few minutes, the liquid also gradually becoming coloured. If no formaldehyde is present, the liquid will at once turn brown, rapidly changing to black. The test is sensitive to a dilution of at least 1 part formaldehyde to 250,000 parts milk, and is given even more rapidly by milk which has stood for several hours than by a fresh sample. A. G. L.

Detection of Cocoanut Oil in True Butter. F. Ranwez. (*Ann. Pharm.*, 1901, vii., 241; through *Chem. Zeit. Rep.*, 1901, 240.)—Vandam has shown that the amount of acids, soluble in alcohol at 60°, contained in cocoanut oil, is much greater than that in true butter or in margarine. Expressed as c.c.s of decinormal potash, and calculated upon 5 grammes of the sample, five specimens of butter have given 10·3 to 11·1; cocoanut oil, 44·2; and margarine, 3·6. If the proportions of these acids insoluble in water, and those soluble in alcohol at 60°, are expressed in the same manner—*e.g.*, butter, 4·6 to 5·2; cocoanut oil, 42·0; margarine, 3·1, these differences become much larger. Therefore an addition of 12 or 13 per cent. of cocoanut oil to cow's butter would double the amount of water-insoluble acids in the alcohol-soluble acids, while an addition of 25 per cent. thereof would treble it. F. H. L.

The Determination of Starch in Cereals. L. Lindet. (*Journ. Pharm. Chim.*, 1901, xiv., 397-400.)—In order to obviate the difficulty of filtration in his method (ANALYST, xxii., 20), and to prevent cellular débris being weighed with the starch, the author proposes the following modification.

The starch washings passing through the sieve after the digestion with pepsin and hydrochloric acid are collected in a 500 c.c. conical flask, where the starch is allowed to subside, and is washed once or twice by decantation. As the starch settles but slowly, it is necessary to allow the liquid to stand from twelve to twenty-four hours after each washing, any bacterial action being prevented by the addition of a little formaldehyde.

The washed starch is then heated for an hour at 110° C. with 100 c.c. of 0·25, 0·5, or 1 per cent. sulphuric acid, and the resulting saccharified solution neutralized and diluted to 250 c.c. The amount of glucose and of dextrin are then determined by reduction with Fehling's solution and polarization, and the original amount of starch calculated from the results.

When much oil is present in a cereal it is advisable to extract it prior to the digestion with pepsin, since otherwise the starch does not separate well in the decantation.

C. A. M.

Some Analyses of Bulgarian Buffalo and Sheep Butter, Lard, and Walnut Oil. N. Petkow. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1901, iv., 823, 827.)—The maximum, minimum, and average results of the analyses of samples of buffalo and sheep butter are given below. It will be seen that the composition of these does not differ greatly from that of cow's butter, although the Reichert-Meissl figure for buffalo butter is somewhat higher. The average amount of water in the buffalo butters was 14·39 per cent. and in the sheep butters 12·72 per cent.

BUFFALO BUTTER (14 samples).

	Specific Gravity at 100° C.	Refractometer Number at 40° C.	Reichert-Meissl Number.	Köttstorfer Number.	Iodine Number.	Insoluble Fatty Acids. Per Cent.	Free Acidity, ° Burstyn.
Maximum ...	0·8700	44·8	40·10	234·7	45·52	87·20	6·13
Minimum ...	0·8680	43·8	30·90	222·5	30·29	89·20	3·22
Average... ...	0·8692	44·2	34·20	229·0	36·75	88·19	4·51
SHEEP BUTTER (12 samples).							
Maximum ...	0·8695	45·5	29·37	234·0	38·48	89·90	3·21
Minimum ...	0·8690	42·5	23·21	223·0	29·70	87·90	0·50
Average... ...	0·8693	44·4	26·68	227·8	35·14	88·50	2·01
LARD (3 samples).							
Maximum ...	0·861	52·0	0·52	198·8	65·56	96·14	1·08
Minimum ...	0·860	51·8	0·55	198·2	65·23	96·01	1·02
Average... ...	0·860	52·0	0·53	198·4	65·36	96·08	1·05
WALNUT OIL (5 samples, cold pressed).							
	At 15° C.						
Maximum ...	0·9260	68·0	—	—	148·43	—	5·97
Minimum ...	0·9255	67·0	—	—	147·92	—	3·17
Average... ...	0·9258	67·7	—	—	148·21	—	4·38

W. P. S.

Prussic Acid in Sweet Cassava. Carmody. (*The Lancet*, September, 1900.) —The author confirms the statement of Francis (ANALYST, 1877, 4-7) that prussic acid is present in sweet cassava to a considerable extent. He finds that the prussic acid is not uniformly distributed throughout the tuber, the inner part containing on the average 0·006 per cent., whilst the skin and outer cortical layer contain 0·028 per cent. of HCN. In bitter cassava the prussic acid is uniformly distributed, the average amount being 0·023 per cent.

W. P. S.

The Decomposition of Food Materials by Micro-organisms. Part I.: Fat-consuming Micro-organisms. J. König, A. Spieckermann, and W. Bremer. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1901, iv., 721-744, and 769-780.)—Three samples of cottonseed meal were used in the experiments described in this paper. They contained fungus spores, as well as bacteria of the hay and potato species. The fungi only increased in growth when the percentage of water in the meal was not under 14 per cent. From 14 to 30 per cent. of water was most favourable to the growth of the fungi, whilst with above 30 per cent. the bacteria obtained the upper hand. The mould flora changed with an increase of moisture. *Eurotium repens* first appeared, soon followed by *Eurotium rubrum*; with 20 per cent. of moisture the "Oïdium" species of mould-fungi were noticed, and with 25 per cent. *Penicillium glaucum*. The growth of the fungi was always accompanied with a loss of organic matter, but an increase of water. On the appearance of *Penicillium glaucum*, the fat and non-nitrogenous extractives were rapidly attacked, and also the pentosans to a small extent. A small quantity of proteid matter was converted into water-soluble organic bodies, but no ammonia was formed. The nitrogenous matter was acted on to a small degree with the liberation of nitrogen. The bacteria obtained the carbon they required from the non-nitrogenous extractives (raffinose, etc.) and pentosans, decomposing the fat only to a small extent. On the other hand, the proteids were decomposed with the production of ammonia. Experiments with pure cultures of the fungi on sterilized cottonseed meal, and also on culture media containing fat, showed the same results as above. From cultures of *Aspergillus flavus* and *Eurotium repens* on sterilized cotton meal, an enzyme was extracted by means of glycerin, which had the property of forming butyric acid from monobutyrin. This enzyme did not appear to act on cotton oil, but the fact that the amount of fatty acid increased in the experiments points to some decomposition of the higher glycerides. The fat is apparently for the most part directly converted into carbon dioxide and water by the action of the fungi. W. P. S.

Detection of Cherry Juice in Raspberry Juice and Red Wines. K. Windisch. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1901, iv., 817-825.)—All cherries examined by the author contained hydrocyanic acid (from amygdalin). This acid is present in the flesh of the cherries as well as in the stones, although in smaller quantity. The fermented juices of cherries, both with and without stones, always contained hydrocyanic acid. The presence of hydrocyanic acid serves as a means for detecting cherry juice in other juices. The test is applied as follows: 20 to 30 c.c. of the fruit juice are distilled until about 2 c.c. have come over. To the latter is added a drop of guaiacum tincture, together with a drop of dilute copper sulphate solution. The appearance of a blue colour indicates the presence of hydrocyanic acid. The colour is fugitive, and if very faint may be intensified by shaking out with a little chloroform, in which it dissolves. A negative test does not prove the absence of cherry syrup, as the original amount of hydrocyanic acid in the cherries may have been very small. W. P. S.

Estimation of Morphine in Opium by means of Ammoniacal Silver Chloride. C. Reichard. (*Chem. Zeit.*, 1901, xxv., 816.)—If the opium sample exists as an alcoholic solution, it is diluted with three times its volume of water and evaporated to its original bulk; otherwise, about 2 or 3 grammes of the material are extracted for an hour with ten to twenty times that amount of boiling water, shaking or mechanically agitating the while. The mass is filtered, and the residue washed several times with boiling water. To the filtrate and washings a small excess of ammoniacal silver chloride is added, and the whole is warmed gently. After standing some time, when the precipitate no longer increases and the liquid is clear, the deposit is collected, washed till free from dissolved silver compounds, dried, and ignited in porcelain to give a button of metallic silver. Two atoms of silver correspond with one of morphine. Experiments show that the other opium alkaloids and constituents do not reduce ammoniacal silver; but to insure absence of (adventitious) reducing matter, another portion of the sample should be extracted with water, precipitated with ammonia to throw down all the alkaloids, and the filtrate examined for any reducing power. Since the reagent contains excess of ammonia, its first effect is simply to precipitate all the alkaloids in the opium, so that the substance on the filter is a mixture of metallic silver with the other opium bases; but inasmuch as the latter are all destroyed by the ignition, this does not affect the accuracy of the process.

F. H. L.

The Estimation of Mercury in Antiseptic Solutions. G. Meillère. (*Journ. Pharm. Chim.*, 1901, xiv., 356-359.)—When the mercury is present in the form of chloride or iodide, it can be extracted quantitatively by shaking the liquid with an excess of sulphuric ether or acetic ether. Thus, 5 c.c. of a solution of mercuric chloride containing 0·0343 gramme of mercury yielded, on evaporation of the ethereal extract, a residue containing 0·0338 gramme of mercury.

For determining the mercury in the salt thus isolated, the author precipitates the metal in the free state by means of stannous chloride or magnesium and hydrochloric acid, etc., and washes and dries the precipitate in a weighed tube, with the aid of centrifugal force.

Mercuric cyanide is usually in a sufficiently pure state, and is readily determined in its solution, but the oxycyanide, when properly prepared, is much less soluble. Hence the commercial salt is frequently a mixture in varying proportions of mercuric cyanide and oxycyanide.

In various commercial samples the author has found from 76·8 to 81 per cent. of mercury, as against 85·04 per cent. by theory.

The cyanogen can be determined by treating 0·1 gramme of the salt with standard iodine solution, in the presence of an excess of alkali-metal carbonate, and after the lapse of several minutes titrating the liquid with a standard solution of thiosulphate.

C. A. M.

ORGANIC ANALYSIS.

Estimation of Nicotine in Tobacco. J. Toth. (*Chem. Zeit.*, 1901, xxv., 610.) —The sample is dried in the air or over quicklime, ground or beaten to powder, and 6 grammes of it are weighed into a 200 or 300 c.c. porcelain basin. Here it is moistened and well rubbed up with 10 c.c. of 20 per cent. sodium hydroxide, and plaster of Paris is added by degrees till the whole forms a dry powdery mass. (This is better than adding plaster to only a paste-like consistency, as recommended by Neumann.) The powder is brought into a stout 25 × 5 centimetre cylinder, where it is repeatedly agitated for an hour with 100 c.c. of a mixture of ether and petroleum spirit. Twenty-five c.c. of the clear liquid are drawn off, run into a glass basin, treated with 40 or 50 c.c. of water and 1 drop of iodeosin solution; then an excess of decinormal acid is introduced, and the solution titrated back with alkali of equal strength. The results are accurate.

Experiments on the influence of ammonia on this process show (1) that the 25 c.c. of ethereal extract may contain 0·5 milligramme of ammonia as a maximum, but that the quantity is usually less; and (2) that if the sample contained ammonium salts, 98·5 per cent. of the corresponding ammonia would be retained in the gypsum. The method proposed by Keller (Analyst, 1898, xxiii., 235) for removing ammonia from the ethereal solution by a current of air is not trustworthy, since the loss in alkalinity of the liquid and the alkaline vapours in the escaping air are due to slight volatilization of nicotine itself. Moreover, in Keller's process nicotine is retained by the aqueous liquid when it is extracted with the mixed solvents. F. H. L.

Formaldehyde and Sulphuric Acid as a test for Alkaloids. H. Linke. (*D. Pharm. Ges. Ber.*, 1901, xi., 258; through *Chem. Zeit. Rep.*, 1901, 184.)—The above reagent gives the following colour tests with alkaloids—Colchicine: golden yellow, gradually disappearing. Hydrastinine: greenish yellow, gradually disappearing. Strychnine: green-brown on warming. Atropine: brownish, on warming "dirty greenish-grey-brown." Homatropine: brown on warming. Scopolamine: golden yellow to orange, brown-black on warming. Veratrine: yellow-brown, reddish-brown on warming. Digitaline: brick to wine-red, dark wine-red on warming. Morphine: cherry-red, then violet, grey-black on warming. This reaction is very fine and sharp. Apomorphine: violet, then rust-red and black-blue, greenish-black on warming. Codeine: violet-blue, brown-black on warming—a fine and very sharp reaction.

Cocaine, pilocarpine, cantharidine, eserine, and caffeine give no reactions either hot or cold. F. H. L.

A Reaction of Urotropine and Piperazine. Manseau. (*Bull. de la Soc. Pharm.*, Bordeaux, June, 1901; *Ann. de Chim. anal.*, 1901, vi., 339, 340.)—The author's tests are based upon the reaction used by Jorissen for the detection o formaldehyde (Analyst, xxiii., 41).

Urotropine, or hexamethylene-tetramine, is decomposed on treatment with acids into formaldehyde and ammonia, and can thus be detected by the purple coloration, changing to blue, which it then gives with morphine.

By using other opium alkaloids instead of morphine, the following colorations are obtained: codeine, blue changing to dark-green; apomorphine, violet-blue; narceïne, saffron-yellow; thebaine, greenish-yellow; and narcotine, golden-yellow.

Piperazine, or diethylene-diamine, also gives colorations when treated with opium alkaloids in the presence of sulphuric acid. Thus, with morphine and codeine there is a slight violet tint, whilst with papaverine, narceïne, narcotine, and thebaine the colours vary from bright yellow to orange and deep brown. C. A. M.

Action of Reducing Sugars on Nickel Salts in Alkaline Solution. M. Duyk. (*Bull. de l'Ass. belge.*, 1901, xv., 267, 268.)—The author's reagent consists of 25 c.c. of a 20 per cent. solution of nickel sulphate, with 25 c.c. of sodium hydroxide solution (specific gravity 1·33), 3 grammes of tartaric acid, and 50 c.c. of water.

On boiling this reagent with the smallest trace of a reducing sugar, a pronounced turbidity is produced, which eventually subsides as a black precipitate of, probably, a sub-oxide of nickel.

The advantages claimed for this reagent over Fehling's solution are that spontaneous reduction does not occur, and that the reaction is more distinct in doubtful cases. Moreover, it is not affected by normal urine, which often decolorizes Fehling's solution and its modifications.

The author has found that cobalt salts are not reduced under the same conditions, and points to the possibility of basing a method of separating nickel from cobalt on this fact. C. A. M.

Isolation of Amido-Sugars. H. Steudel. (*Zeits. physiol. Chem.*, 1901., xxxiii., 223; through *Chem. Zeit. Rep.*, 1901, 275.)—In alkaline solution phenyl isocyanate combines with glucosamine to form a substance which is very insoluble in water, and which separates from its solution in dilute acetic acid as a heavy crystalline powder having a sharp melting-point. When 2·25 grammes of glucosamine hydrochlorate were dissolved in 30 c.c. of water and 10 c.c. of normal potassium hydroxide and 1·19 grammes of phenyl isocyanate dropped in with constant agitation and cooling, the liquid quickly solidified to a thick paste. The solid matter was removed and dissolved in hot acetic acid. On cooling large rhombic crystals separated, which were recrystallized from water and dried at 110° C. On analysis they gave figures corresponding with $C_{13}H_{16}N_2O_5$. This body begins to turn brown at 200°, and melts sharply at 210° C. Isoglucosamine yields a similar product, and the author believes that other amido-sugars may be isolated by the same process. F. H. L.

Detection of Mineral Oil in Rosin Oil. D. Holde. (*Mittheil. Kgl. Techn. Versuchsanstalten zu Berlin*, 1901, xix., 39; through *Chem. Zeit. Rep.*, 1901, 232.)—Ten c.c. of the oil are dissolved in 90 c.c. of 96 per cent. alcohol at the laboratory temperature, shaking if necessary. If a large quantity of oil remains undissolved, it may be considered as a sign of the presence of much mineral oil. This may be confirmed by collecting the oil, rinsing it with a little alcohol, and determining its refractive index. If, however, nearly all the original oil dissolves in the spirit, any undissolved matter is neglected. The whole is cautiously diluted with water till a distinct milkiness is produced, and the mixture is set aside (all night if so required). The clear alcoholic layer is poured off from the residue, which should not exceed 1 c.c., and the oil is rinsed with a few c.c. of alcohol. It is then dissolved as before in 20 c.c. of 96 per cent. alcohol, and treated with water till not exceeding 3 drops of oil separate. These are again rinsed with spirit, washed with hot absolute alcohol, dried in a basin, and examined for their refractive index. An index of below 1·5330 at a temperature between 15° and 18° C. shows the presence of mineral oil.

F. H. L.

Linseed Oil and its Adulterants. P. C. McIlhiney. (*Report to the Commissioner of Agriculture of New York State*, 1901, 1-31.)—The author states that the character of linseed oil now made in New York State does not correspond with that given in even recent text-books, for it is prepared by heating metallic oxides with a small quantity of the oil, and adding this "drier" to the main body, which is kept at a much lower temperature than in the old-fashioned method.

The figures given by the oil treated in the new way are practically identical with those for raw linseed oil.

A table is also given showing the corrections for temperature to be added to or subtracted from the readings of a glass hydrometer correct at 15·5° C. when immersed in linseed oil. According to this, the correction for 1° F. averages 0·000361, and for 1° C., 0·000650.

Hehner and Mitchell's test, based on the amount of insoluble bromine derivatives (ANALYST, xxiii., 310), is regarded as a valuable one for the detection of the adulteration of linseed oil. The author has obtained results substantially in agreement with theirs.

C. A. M.

The Detection of Drying and Marine Animal Oils. G. Halphen. (*Journ. Pharm. Chim.*, 1901, xiv., 359-365 and 391-397.)—The author's test is based upon the fact recorded by Hehner and Mitchell (ANALYST, xxiii., 315) that these oils (*i.e.*, the glycerides) yield insoluble derivates on treatment with bromine.

His reagent is prepared by mixing twenty-eight parts by volume of glacial acetic acid with four parts of nitro-benzene and one part of bromine; 0·5 gramme of the oil under examination is shaken in a stoppered tube, with 10 c.c. of this reagent, until the liquid is homogeneous, and is then allowed to stand.

In this way it was found that no turbidity resulted after an hour in the case of olive, almond, castor, cotton-seed, poppy, lard, and neatsfoot oil.

With the following oils no precipitate was formed, but only a slight turbidity: Jaffa sesame oil, and certain samples of the foot oil of the horse and sheep.

A marked turbidity, and eventually a heavy precipitate, were given by the following: nut oil (precipitate after five to ten minutes), hempseed, linseed, seal, cod-liver, whale, spermaceti, and Japanese fish oils.

Colza oil was distinguished by giving a turbidity which on standing separated into two layers of liquid.

The precipitates thus obtained with the different classes of oils vary in their properties. Thus, on washing the insoluble bromides of linseed oil and of fish oils with sulphuric ether to remove the excess of the reagent, and then pressing them between filter-paper and drying them in the air, heavy substances are left, which differ in their behaviour towards carbon tetrachloride.

The solutions, obtained by treating the precipitates with this solvent in the proportion of about 2·5 c.c. to each 0·1 gramme at a temperature near the boiling-point and then filtering, are very different. The linseed oil precipitate yields a perfectly clear solution, which, on cooling, deposits, little by little, a gelatinous precipitate. With fish oils, on the other hand, the solution is opalescent, and only deposits a trace of a crystalline precipitate on cooling.

The bromides of other drying oils behave like linseed in this respect, but in the case of hemp-seed oil several hours are required before the gelatinous precipitate is deposited.

The other marine animal oils mentioned above all yielded opalescent solutions in this test.

In the case of the foot oil of terrestrial animals, a clear solution, which does not yield a gelatinous precipitate, is obtained.

The author describes a series of test experiments to show that by this method it is possible to detect 10 per cent. of linseed, hempseed, whale, or fish oil in castor, cotton-seed, or olive oils. C. A. M.

Occurrence of Mixed Glycerides in Cacao Butter. J. Klimont. (*Berichte*, 1901, xxxiv., 2636.)—Cacao butter, in the author's experiments, was separated into three fractions by crystallization from acetone. The first of these melted at 64° C., had an iodine value of 0, after repeated recrystallization, and finally yielded crystals melting at 70° C. It was found to consist of a mixture of tripalmitin and tristearin.

The second fraction, consisting of granular crystals, melted between 31° and 32° C., and had an iodine value of 28·9, a saponification value of 196·4, and a composition agreeing with the formula $C_{55}H_{104}O_6$. The author concluded that it was a triglyceride containing the three radicles of palmitic, oleic, and stearic acid.

The third fraction was also regarded as a mixed glyceride. It melted at 26° to 27° C., had an iodine value of 31·7, and a saponification value of 210·5, and from an elementary analysis was concluded to have the composition $C_{51}H_{96}O_6$.

The author was unable to identify triolein in cacao butter. C. A. M.

The Use of Iodine Monobromide in the Analysis of Fats and Oils. J. Hanus. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1901, xx., 913-920.)—For determining the iodine absorption of fats and oils the author uses iodine monobromide. This is prepared by slowly dropping 13 grammes of bromine into 20 grammes of finely powdered iodine contained in a beaker. During the addition of the bromine (which should take about ten minutes) the contents of the beaker are well stirred and kept below 8° C. Each drop of bromine causes a lump to form which must be broken up. When all the bromine has been added, a rapid stream of carbon dioxide is passed through the beaker, to remove excess of bromine. The iodine monobromide so obtained is a dark-grey substance, having a metallic lustre. It keeps well in closed vessels, and is soluble in ethyl alcohol and glacial acetic acid. The reagent is prepared by dissolving 10 grammes of the monobromide in 500 c.c. of glacial acetic acid. The exact strength of this solution is determined by titrating 10 c.c. after the addition of 15 c.c. of 10 per cent. potassium iodide solution, with standard thiosulphate solution.

To obtain the iodine number of an oil or fat, from 0·1 to 0·7 gramme of the same is dissolved in 10 c.c. of chloroform; 25 c.c. of the above reagent are added, and the mixture is allowed to stand for fifteen minutes. Fifteen c.c. of 10 per cent. potassium iodide solution are then added, and the titration carried out as usual with thiosulphate solution. The use of starch solution at the end of the titrations may be dispensed with. With oils and fats having an iodine absorption below 100, the reaction is complete in ten minutes. The strength of the reagent should be checked in each set of determinations, as it slightly decreases. The results obtained agree well with those of the Hübl method. W. P. S.

Quantitative Separation of Cholesterin (Phytosterin) from Fats. E. Ritter. (*Chem. Zeit.*, 1901, xxv., 872.)—About 50 grammes of the fat are weighed into a 1,500 c.c. porcelain basin, using, if necessary, 100 c.c. of alcohol to rinse it out of the weighing vessel. It is then heated to the boiling-point on the water-bath, and mixed, with continual stirring, with an alcoholic solution of sodium ethoxide, made by dissolving 8 grammes of sodium (freed from petroleum and oily matter) in 160 c.c. of 99 per cent. alcohol without cooling. The whole is heated on the water-bath till the alcohol has evaporated, when 75 grammes (one and a half times the weight of the fat taken) of common salt are added together with enough water to dissolve most or all the contents of the basin. With constant stirring the liquid is evaporated to dryness, first over a naked flame, next on the water-bath, then in a drying oven at 80° C., and finally in the desiccator over sulphuric acid, beginning to reduce the mass to powder as soon as possible. The residue is extracted with ether in a large Soxhlet, employing a fat-free paper thimble with a plug of cotton-wool on top. This operation is generally finished in about nine hours. The ether in the flask is usually turbid at first owing to traces of glycerin, but these gradually adhere to the walls and remain attached, when the ether is poured off into another vessel to make room for a fresh quantity. Finally, all the ethereal extract is brought into a 750 or 1,000 c.c. flask, the solvent distilled away, the residue taken up in the minimum of alcohol, and water is added nearly to fill the flask. The precipitated substance is collected on a paper, washed with water, dried on the filter in an oven at 60° C., scraped off into a tared basin, the remainder rinsed off the paper with ether, and the whole is distilled or evaporated, dried at 100° or 120° C., and weighed. F. H. L.

Characteristics of Neroli Oil from different Sources. E. Theulier. (*Bull. Soc. Chim.*, 1901, xxv., 762-764.)—The author has examined thirty-three specimens

oil distilled, by the method in use at Grasse, from orange-flowers collected
May 16 and June 2 of the present year.
as found that the specific gravity varied but little whatever the origin of
he extreme values being 0·869 and 0·8726, and the mean 0·8709.
differences in the rotatory power were somewhat greater, ranging from
to +7° 20′ in a 100 millimetre tube at 23° C. The mean was +4° 48′.
proportion of methyl anthranilate was determined by the method of A.
d O. Zeitschel, in which this ester is precipitated in the form of a sulphate
only soluble with difficulty in ether, but which can be dissolved in water
ted. The average quantity in the samples of oil examined was 0·7 per cent.
esters determined as linalyl acetate varied between 8·08 and 14·7 per cent.,
n being 11·27 per cent. This maximum was lower than in preceding years,
the highest proportion was usually 17 to 18 per cent.
yield of oil obtained from the flowers varied considerably with their origin.
d from 0·91 to 1·32 per cent. C. A. M.

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nposition of Cannes Geranium Oil. **Jeancard and Satie.** (*Bull. Soc.*
1901, xxv., 516-519.)—In continuation of their research on geranium oil
T, xxv., 160) the authors have made a complete examination of thirty-one
of Cannes oil distilled at different periods between September 3 and
6.

the subjoined table their results are summarized, together with those of the
residual oils after neutralization, saponification, and determination of the
:

m Oil.	Specific Gravity at 25° C.	Rotatory Power (l=100) at 15° C.	Superficial Tension.	Specific Viscosity.	Saponification Value.		Solubility in 70 per cent. Alcohol at 15° C. 1 vol. in
					Hot.	Cold.	
y ...	0·8874 to 0·8906	−9°38′ to −10°55′	2·939 to 2·970	1′18″ to 1′25″	49 to 54	27 to 36	1·7 to 1·9
ized	0·8777	−8°50′	2·910	1′23″	16·8	0	1·0
ted ...	0·9070	−2°40′	3·042	0′39″	211·4	0	Insol. in 20
ed ...	0·8802	−3°0′	2·907	1′42″	0	0	1·5

m these results the authors conclude that: (1) Neutralization diminishes
cific gravity, rotatory power and surface tension, but increases the solubility;
ylation increases the specific gravity and surface tension, but diminishes the
viscosity and solubility; (3) saponification increases the specific viscosity and
bility.

e fact that neutralization increases the solubility explains why Cannes
m oil is somewhat more soluble than geranium oils from other sources, for it
less free acids than the others. C. A. M.

Myrcenol. Barbier. (*Comptes Rend.*, cxxxii., 1048; through *Pharm. Jour* 1901, lxvii., 89.)—The alcohol obtained by the hydration of myrcene is not identi with licareol, as was stated by Power and Kleber, but is a new alcohol—myrcenol having the constitutional formula:

$$(CH_3)_2C : CH{\cdot}CH_2{\cdot}C(CH_3){\cdot}OH{\cdot}CH{\cdot}CH_2.$$

It is a colourless, strongly-smelling liquid, boiling at 99° to 101° C. at 10-millimet pressure, and having a specific gravity of 0·9072 at 14·5° C. The formula give above is identical with that of Tiemann for licareol (linalool), and consequently th formula of the latter needs reviewing. The acetate—a pungent, oily liquid—has a entirely different odour from that of linalyl acetate. By oxidation with chrom acid, an aldehyde, $C_{10}H_{16}O$, is obtained, which is not citral. It boils at 110° C. a 10-millimetre pressure, gives an oxime boiling at 148° to 150° C. under the sam pressure, and a semi-carbazone melting at 195° to 196° C. W. P. S.

On the Bromine and Iodine Values of Proteids. W. Vaubel. (*Zeit. an Chem.*, 1901, xl., 470-474.)—It was shown by Blum and the author (*J. prakt. Chem* 1898, lvi., 393; and lvii., 365) that undecomposed albuminous bodies, such as eg albumin and casein, were capable of absorbing the following amounts of halogens the maximum: Iodine, 6 to 7; bromine, 4 to 5; chlorine, 2 to 3; and fluorine, abo 1 per cent. It was also found that in these experiments a greater quantity of hydr chloric acid., etc., was liberated than corresponded to the degree of substitution. Th author has therefore made experiments to determine the total amount of haloge used, both in the substitution and in the formation of hydrobromic or hydriodic aci by the withdrawal of hydrogen. Using a solution of bromate for the titration, an deducting 9·0 grammes of bromine for the substitution and the corresponding hydro bromic acid liberated, the following results were obtained calculated on 100 grammes of the anhydrous and ash-free material:

	Bromine.
Egg-albumin	35·04 grammes.
Blood-albumin	40·76 ,,
Casein	27·00 ,,

On dissolving these proteids in alkali, and boiling them for an equal length o time, the sulphur, split off as hydrogen sulphide and reacting with the bromine, was eliminated and less bromine absorbed. This sulphur as hydrogen sulphide was calculated from the bromine results to be as follows: Egg-albumin, 1·12; blood albumin, 0·72; and casein, 0·45 per cent.

In determining the iodine values Dieterich's method was first used. On gramme of the air-dried albumin, etc., was treated with 50 c.c. of water in a stoppere litre-flask, and, after standing over night, mixed with 20 c.c. of $\frac{N}{10}$ iodine solution and left for three days; 500 c.c. of water were then added, and the liquid titrated wit $\frac{N}{10}$ thiosulphate solution, of which not more than 11 c.c. or less than 6·5 c.c. wa required.

In this way it was found that the anhydrous and ash-free substances consume the following amounts of iodine: Egg-albumin, 21·345; blood-albumin, 22·285; and casein, 20·844 per cent.

Deducting 6·5 × 2 grammes for the iodine absorbed by substitution and the corresponding hydriodic acid liberated, the following values were obtained for the hydrogen withdrawn by the iodine without substitution:

	Iodine.	Hydrogen.	Bromine.	Hydrogen.
Egg-albumin	8·345	0·0657	35·04	0·4380
Blood-albumin	9·285	0·0731	40·76	0·5095
Casein	7·644	0·0601	27·00	0·3450

From a comparison with the bromination figures, these iodine values appear to be incomplete.

By treating the solution of the proteid with 10 grammes of sodium bicarbonate, and adding 100 c.c. of the iodine solution, considerably higher results were obtained, thus: Egg-albumin, 56·41; blood-albumin, 60·87; and casein, 51·88 per cent. Deducting from these values 13 grammes of iodine for the substitution process, the following figures were obtained, in which the amounts of hydrogen are in much closer agreement with the bromination results:

	Iodine. Per cent.	Hydrogen. Per cent.
Egg-albumin	43·41	0·3418
Blood-albumin	47·87	0·377
Casein	38·88	0·3061

C. A. M.

Gas-Volumetric Estimation of Chlorides and Phosphates in Urine. **E. Riegler.** (*Wiener med. Bl.*, 1901, xxiv., 527; through *Chem. Zeit. Rep.*, 1901, 246.)—This process depends on the liberation of nitrogen from hydrazine sulphate by means of silver chloride, according to the equation

$$4AgCl + N_2H_4.H_2SO_4 + 6NaOH = 4Ag + 4NaCl + Na_2SO_4 + 6H_2O + N_2,$$

the nitrogen being measured in any convenient instrument and converted into weight by Baumann's tables. From the chlorides of the urine the silver chloride is prepared in the usual fashion; the phosphoric acid is thrown down with magnesia mixture, the precipitate filtered and washed, and dissolved in nitric acid. The solution is mixed with silver nitrate and ammonia to a faintly alkaline reaction, and boiled for a few minutes. The silver phosphate is then decomposed with hydrochloric acid in nitric acid solution. One milligramme of nitrogen corresponds with 8·23 milligrammes of NaCl, or with 3·34 milligrammes of P_2O_5.

F. H. L.

The Determination of Traces of Sugar in Urine. **E. Raimann.** (*Zeit. anal. Chem.*, 1901, xl., 390-402.)—In this paper the author first shows that a single precipitation of the sugar with phenylhydrazine gives unreliable results, and then describes the following modification: 500 c.c. of the urine are fermented with fresh yeast at 34° C. for twenty-four hours, and then treated at the same time as 500 c.c. of the non-fermented urine in the following manner: After the addition of 100 c.c. of a 25 per cent. solution of lead acetate, the liquid is filtered, and the excess of lead removed from the filtrate by means of hydrogen sulphide; 450 c.c. of the second filtrate are evaporated on the water-bath to about 100 c.c., then made up to 120 c.c. and filtered. Of this filtrate 100 c.c. are heated on the water-bath for one and a half hours with 5 c.c. of glacial acetic acid and 3 c.c. of phenylhydrazine, and, after standing for twenty-four hours, the precipitate collected on a dried and weighed filter-paper, and dried first in the air and subsequently at 110° C. until constant in weight.

The difference between the weight of osazone thus obtained and that given by the unfermented urine is taken as pure phenyl-glucosazone.

In test experiments in which known quantities of sugar were added to the fermented urine the amount of osazone calculated into glucose only averaged 49 per cent. of the theoretical quantity, and in order to obtain the percentage of sugar in the urine the difference in weight of the osazone precipitates was multiplied by the factor 0·329.

In this way the following results, among others, were obtained:

Sugar Added. Per cent.	Sugar Found. Per cent.	Error. Per cent.
0·005	0·006	+ 0·001
0·100	0·095	− 0·005
0·206	0·213	+ 0·011

The fermented urine invariably yields with phenylhydrazine a non-crystalline precipitate, which adheres firmly to the bottom of the beaker. C. A. M.

The Detection of Acetanilide in Urine. A. Petermann. (*Ann. de Chim. anal.*, 1901, vi., 165.)—About 10 c.c. of the urine are boiled for a few minutes with 25 c.c. of hydrochloric acid. After cooling, 1 c.c. of a 3 per cent. aqueous solution of phenol is added, followed by 2 or 3 drops of a 10 per cent. solution of calcium chloride, the liquid being shaken after the addition of each drop. In the presence of para-amido-phenol, the decomposition-product of acetanilide, the urine acquires a red colour, which, however, it is difficult to distinguish from the coloration which normal urine gives with hydrochloric acid.

Concentrated ammonium hydroxide is next poured down the side of the glass, this reagent changing the red colour to blue. The reaction is certain in the presence of acetanilide, but may be doubtful when it is not present.

As a confirmatory test 100 or 200 c.c. of the suspected urine are mixed with a fourth of its volume of hydrochloric acid, and boiled for several minutes. When cool, the liquid is neutralized with calcium carbonate and extracted several times with ether. The latter is decanted, and shaken with water containing one-fourth of its volume of hydrochloric acid. The lower layer is then withdrawn, gently warmed to expel the last traces of ether, and tested for acetanilide as above. C. A. M.

Estimation of Ammonia in Urine. O. Folin. (*Zeits. physiol. Chem.*, 1901, xxxii., 515; through *Chem. Zeit. Rep.*, 1901, 239.)—The urine, or the solution of urea, is diluted with 400 or 500 c.c. of water, and distilled with magnesia or lime-water for forty-five minutes (counting only the time of actual ebullition), collecting the distillate in decinormal acid. Without stopping the operation, a volume of water, roughly equal to that which has distilled over, is then added to the retort, and the process is continued for a second period of forty-five minutes, using a different receiver. On the assumption that the decomposition of the urea proceeds at a uniform speed, the amount of ammonia in the second distillate (as determined by titration), deducted from that in the first, gives the ammonia which existed as such in the urine.

F. H. L.

On the Estimation of Urea in Urine. J. H. Long. (*Journ. Amer. Chem. Soc.*, xxiii., 633.)—In the opinion of the authors, the old Liebig method, in which the urea is titrated with mercuric nitrate solution, is capable of rendering good service where a number of quick comparative determinations are to be made. The method was discarded because other substances besides urea present in urine react with mercuric nitrate, and, consequently, the authors have made numerous experiments to determine the influence which these bodies exercise. The most important of these, after the chloride—the effect of which is well known and easily corrected—are ammonia, uric acid, and creatinin. The average correction for these three bodies was found to be 2·0 c.c. mercury solution, which may be used in the case of fresh urine with only a small margin of error, whilst, if the disturbing bodies themselves are estimated, the corresponding correction may be applied with considerable accuracy.

A. G. L.

Estimation of Urea in Urine. O. Folin. (*Zeits. physiol. Chem.*, 1901, xxxii., 504; through *Chem. Zeit. Rep.*, 1901, 239.)—Urea is quantitatively decomposed into ammonia and carbon dioxide within half an hour when it is heated with magnesium chloride boiling in its own water of crystallization (at 160° C.). Accordingly, 3 c.c. of the sample, 20 grammes of magnesium chloride, and 2 c.c. of strong hydrochloric acid, are boiled in a 200 c.c. Erlenmeyer flask under a 200 × 10 millimetre reflux tube till the drops of condensed liquid fall with a hissing noise into the flask. The boiling is continued for another twenty-five or thirty minutes, when the whole is cautiously diluted with water, rinsed into a 1-litre flask, and distilled with 7 c.c. of 20 per cent. sodium hydroxide till about 350 c.c. have passed over. The distillate is boiled up, cooled, and titrated, every 1 c.c. of decinormal ammonia in the liquid corresponding with 3 milligrammes of urea. Corrections must be made for any ammonia in the magnesium chloride, and for pre-existing ammonia in the urine.

F. H. L.

The Analysis of Shellac. E. J. Parry. (*Chemist and Druggist*, 1901, 689.)—The writer states that samples of shellac absolutely free from rosin are the exception and not the rule. For detecting this adulteration with rosin, he relies on the determination of the acid number, the ester value, and the iodine absorption. Pure samples of shellac gave acid values between 55 and 65, with an average of 60, whilst rosins gave an average of 160. The ester values of shellacs lie between 155 and 175, those of rosins generally below 20, averaging about 10. The iodine numbers of

different samples of shellac examined lay between 4 and 10. Rosin gives an average iodine absorption of 110.
W. P. S.

Detection of Chrome Yellow in Dyed Cotton. P. Cazeneuve. (*Bull. Soc. Chim.*, 1901, xxv., 761-762.)—Diphenyl carbazide is an extremely sensitive reagent for chromic acid, the limit of sensibility exceeding 1 part in 1,000,000.

The cotton fibre is treated with 1 c.c. of a 10 per cent. solution of potassium hydroxide, an immediate decolorization being effected in the presence of lead chromate. The liquid is then rendered strongly acid with acetic acid, and shaken with a pinch of diphenyl carbazide or its acetate, when a pronounced violet colour confirms the presence of chromic acid.
C. A. M.

The Use of Certain Indicators by Artificial Light. A. Kufferath. (*Zeits. f. angew. Chem.*, 1901, 916.)—Experiments were carried out to ascertain whether the indicators could be relied upon for use with different sources of artificial light. The indicators used were: Methyl orange, fluorescein, cochineal, coralline, p-nitrophenol, alizarine green B, resazurine, and luteol. These were all tried by daylight, with electric glow-lamp, incandescent gas, and acetylene. From the results it appears that acetylene is the best artificial light to use where it is necessary to distinguish between two different colours, as with methyl orange, cochineal, coralline, alizarine green B, and resazurine. The sort of light is of less importance where the change is of a different nature, as with p-nitrophenol and luteol, which change from colourless to light yellow, and fluorescein, which changes from light yellow to a yellowish-green fluorescence.
A. M.

The Hübl Iodine Solution. M. Kitt. (*Chem. Zeit.*, 1901, xxv., 540.)—The gradual disappearance of active iodine from the ordinary Hübl iodine solution depends on the progress of a chemical reaction which continues until equilibrium is attained. By suitable treatment of the liquid, the author finds it possible to hasten that progress, and to produce a reagent that is far more permanent in strength. Thirty grammes of iodine and 25 grammes of mercuric chloride are dissolved separately in 500 c.c. of 98 per cent. (by weight) alcohol, and the mixed solution is boiled on the water-bath under an inverted condenser for one hour. Before ebullition, the liquid contained 0·7656 gramme of iodine per 25 c.c.; after, 0·49321 gramme. In four days the boiled reagent only lost 0·75 per cent. in strength; in thirty-nine days, 8·12 per cent. Ordinary Hübl iodine loses 2·12 per cent. in one day, and 12·75 per cent. in twenty-seven days.
F. H. L.

The Estimation of Hydrogen Sulphide in Coal Gas. A. Muller. (*Journ. f. Gasbel.*, xliii.; *Chem. News*, lxxxiii. (2163), 217.)—The reagents required are a solution containing 25 grammes of cadmium acetate and 200 c.c. of acetic acid per litre, and a second containing 80 grammes of copper sulphate and 175 c.c. of concentrated sulphuric acid per litre. The gas is passed at a moderate rate through 25 c.c.

he first solution contained in an Erlenmeyer flask, the volume being accurately asured. The contents of the flask are afterwards heated to about 50° to 60° C., the resulting precipitate (CuO) is filtered, washed with warm water, calcined and ghed. Since 1 mol. of CuO corresponds in this reaction to 1 mol. of H_2S, the ime of the latter is found by multiplying the weight of CuO by 34 and dividing the duct by 79—the weight of a litre of hydrogen sulphide at the temperature of the eriment.

C. S.

Determination of the Heating Power of Gases. **W. Hempel.** (*Zeits. f. ew. Chem.*, 1901, 713.)—The general arrangement of the apparatus is shown in . 1. It consists of a burner *A*, which is so arranged that it can be fed with the under examination and at the same time with oxygen. It is thus possible to so

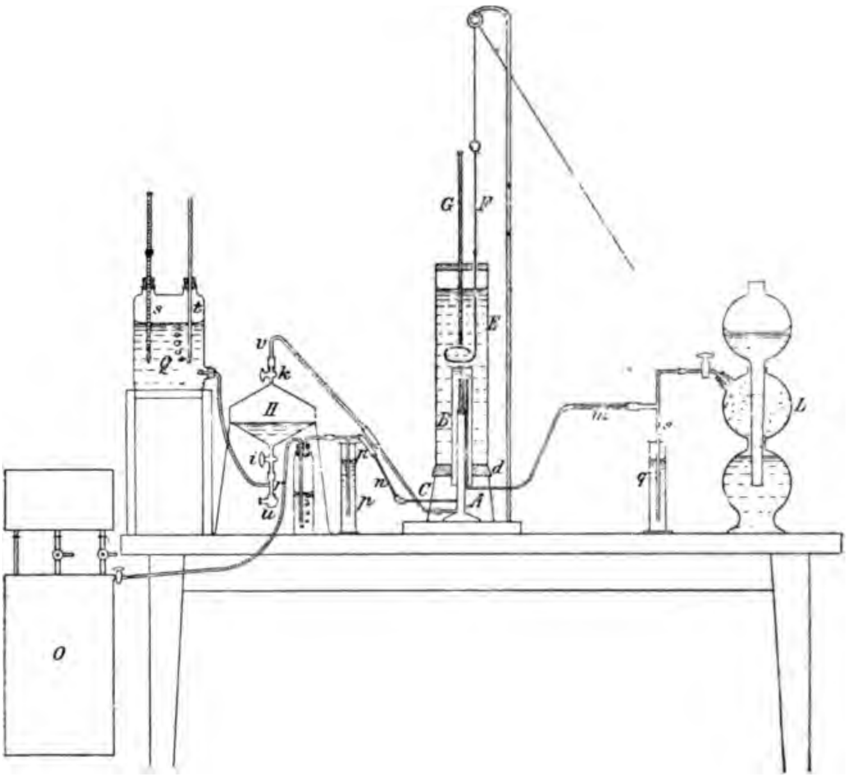


Fig. 1.

reduce the volume of the products of combustion that they are completely led in a copper tube *B* only 18 centimetres long and 3·3 centimetres wide. The per tube is closed above, and is fixed by means of the rubber cork *d* into the le glass tube *E*, in which 500 c.c. water can be placed. The thermometer can read to 0·02° C. The gas is contained in the reservoir *H*, which is made as flat as possible. The gas is ignited by means of a quite small flame of hydrogen, generated in an ordinary generator *L*. The oxygen is contained in the holder *O*.

In order to render it possible to supply exactly the same quantities of hydrogen and oxygen in different determinations, the gases are led through the capillary tubes *m* and *n*, and their flow is regulated so that no bubbles rise out of the cylinders *p* and *q*.

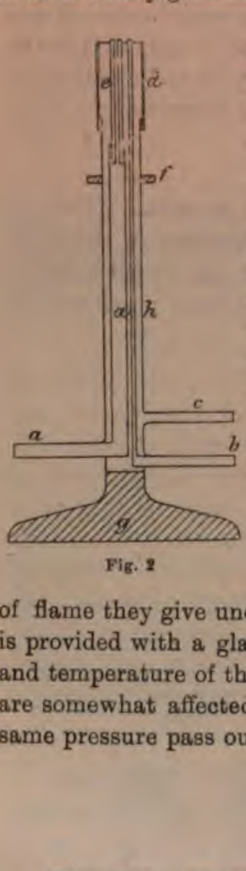


Fig. 2

The construction of the burner is shown in Fig. 2. The gas under examination enters by the tube *a*, hydrogen by the tube *b*, and oxygen by *c*. The tubes are all provided with porcelain tips, *d*, *e*, to prevent conduction of heat. The gas reservoir *H* is connected with the Woulffe bottle *Q*, so arranged that the pressure of the gas remains practically the same throughout. The end of the combustion is indicated by the cessation of the bubbles from *t*. The apparatus is calibrated by burning a reservoir full of pure hydrogen. The small ignition flame is kept burning exactly the same time in each determination; thirty seconds should be long enough. The heating effect of this is determined separately.

For roughly comparative purposes, the relative heating power of gases may be ascertained by observing the length of flame they give under identical conditions. For this purpose a one-hole burner is provided with a glass chimney on which graduations are marked. The pressure and temperature of the gases must, of course, be in each case the same. The results are somewhat affected by the specific gravity of the gas, for light gases under the same pressure pass out more rapidly than heavy ones. A. M.

INORGANIC ANALYSIS.

The Determination of Platinum and Iridium in Platinum Ores. Leidié and Quennessen. (*Journ. Pharm. Chim.*, 1901, xiv., 351-355.)—The following modification of Leidié's method (Analyst, xxvi., 108) is recommended when only the amount of platinum and available iridium in an ore are required: Five grammes of the ore are repeatedly extracted with hot *aqua regia* (nitric acid, specific gravity 1·32, 1 part; hydrochloric acid, specific gravity 1·18, 3 parts) until nothing more dissolves. The extract is evaporated to dryness at 105° to 110° C., and the residue dissolved in a small quantity of water and filtered.

The filtrate is then brought to about 70° C., and treated with sodium nitrite until the liquid becomes neutral to turmeric, when sodium carbonate is added little by little until no more precipitate is formed. The liquid is then filtered, after being heated to boiling, in order to remove the foreign metals.

Sodium hydroxide is now added to the filtrate until the reaction is alkaline, and the osmium and ruthenium expelled as volatile peroxides by means of a current of chlorine, which is passed through the liquid maintained at 70° to 80° C.

The liquid is next neutralized with hydrochloric acid, and any chlorides that may have been formed reconverted into nitrites by the addition of more sodium nitrite. The solution will now only contain alkali salts and the double nitrites of platinum, palladium, iridium, and rhodium.

Estimation of the Platinum.—At this stage in the original method the liquid was saturated with ammonium chloride, but, instead of this, from 30 to 35 per cent. of potassium chloride are introduced. The double nitrites of rhodium and iridium with potassium, which are precipitated, are insoluble in solutions of alkali chlorides.

The nitrites of platinum and palladium in the filtrate are converted into chlorides with hydrochloric acid, and the mass dissolved in boiling water. A slight excess of formaldehyde is now added, and the metals precipitated on boiling the liquid are ignited, reduced in a current of hydrogen, and dissolved in *aqua regia.* The residue left on evaporation of this solution is dissolved in water and treated with a reducing agent, to convert palladic chloride into palladous chloride, after which the platinum can be precipitated with ammonium chloride, and estimated in the usual way.

Estimation of the Iridium.—In a portion of liquid from which the osmium and ruthenium have been expelled, the double nitrites of rhodium and iridium are converted into chlorides by means of *aqua regia* containing but little nitric acid.

The iridium is then precipitated by means of ammonium chloride, and the precipitate washed with ammonium chloride solution, ignited, reduced in hydrogen to the metallic form, and weighed.

The iridium could also be precipitated by means of potassium chloride as the double chloride $2KCl.IrCl_4$. The potassium chloride is removed by washing with water, and the residue reduced, dried at 115° C., and weighed.

The authors point out that this method only gives the available iridium, since any which is present in the free state or in combination with the osmium is insoluble in *aqua regia*, and is left in the residue in the preliminary extraction. C. A. M.

The Estimation of Tin in White-metal Alloys. Frederick Ibbotson and Harry Brearley. (*Chem. News*, 84, 167.)—Tin may be readily estimated by reducing the hot solution of the chloride, cooling in an atmosphere of CO_2, and titrating with iodine and starch. The reduction can be conveniently effected by means of iron, but any excess added must be dissolved completely. If antimony is present, it will be precipitated as metal, and cannot be dissolved again, but as cold acid solutions of stannic chloride are not reduced by antimony, which readily reduces them on heating, the solution may be directly titrated with iodine as usual, very good results being obtained. The antimony may be filtered off after the titration and estimated, but the results obtained are rather low.

An improvement on the above method consists in reducing the stannic chloride with finely-powdered metallic antimony. The reduction of 0·15 gramme of tin is complete after one minute's boiling, and the excess of antimony which remains undissolved acts as a safeguard during the cooling, since it reduces any tin which may have become oxidized whilst the solution is still hot. Cold solutions of stannous chloride take up oxygen less readily. The test analyses given show that the reduction is complete.

The influence of various substances likely to be present in an ordinary analysis on the above method was examined, and it was found that the presence of iron, chromium, nickel, zinc, manganese, aluminium, bismuth, phosphorus, and sulphur is without effect on the results. The quantity of hydrochloric acid present should always be about one-fifth of the total volume. If copper is present, it will be reduced to the cuprous state, but accurate results may nevertheless be obtained if the iodine is added drop by drop to the vigorously agitated solution, so as to prevent the formation of a local excess of iodine. It is also advisable to have rather more hydrochloric acid present, up to about one-third of the total volume. Cobalt apparently gives very slightly high values. Lead is without influence if sufficient hydrochloric acid is present to prevent the formation of lead iodide. The presence of arsenic completely vitiates the results, whether the tin is reduced with iron or with antimony. Mercury is reduced to the metallic state, but is not oxidized in cold solutions. If molybdenum or tungsten is present, a coloured lower oxide is formed, but this is not appreciably reoxidized by the iodine, and the starch blue can be readily distinguished. A. G. L.

The Determination of Sulphur in Iron and Steel. William A. Noyes and L. Leslie Helmer. (*Journ. Amer. Chem. Soc.*, xxiii., 675.)—The authors recommend the following two methods as being more rapid and accurate than those now in use: In the first, 5 grammes of the steel are added in portions at a time to 200 c.c. of water and 8 c.c. of bromine contained in a flask, cooling after each addition. The solution is finally boiled for a moment to expel excess of bromine, filtered, the filtrate added to 130 c.c. of 10 per cent. ammonia contained in a 500 c.c. flask, the liquid made up to the mark, and well mixed. Three hundred c.c. are then filtered off, evaporated in a wide beaker to 100 c.c., and precipitated with 10 c.c. of barium chloride solution, after the addition of 1 drop of hydrochloric acid of specific gravity 1·12. After the precipitate has settled, it is filtered off, ignited, and weighed as usual. The filter-paper containing the original insoluble residue is burnt with some sodium carbonate, the oxidation being completed by adding a little potassium nitrate. The melt is dissolved in water, the solution filtered, acidified with hydrochloric acid, and precipitated with 5 c.c. of barium chloride solution. Three-fifths of the weight of barium sulphate found here is added to the main quantity of barium sulphate found, and the total represents the sulphur in 3 grammes of iron.

In the second method, 5 grammes of the iron, mixed with 7 grammes potassium chlorate, are added in portions at a time to 120 c.c. nitric acid (specific gravity 1·20) and 1 gramme potassium bromide, cooling if necessary. When solution is complete, the liquid is evaporated to dryness in a dish, the residue dissolved in hydrochloric acid and filtered after diluting, both filtrate and residue being treated as in the first method. In the bromine method, the amount of sulphur left in the insoluble residue is larger than in the nitric acid method, but no evaporation to dryness is required, and practically no fumes are given off during solution.

Test experiments, in which known amounts of ammonium ferrous sulphate were added to sulphur-free iron, and to steel containing known amounts of sulphur, gave satisfactory results. The methods also gave concordant and higher results than Blair's aqua regia method, but the values obtained by using the latter are rather irregular. Since the quantities of sulphur left in the residues will probably prove nearly constant for any given class of steel, the authors believe that it will be possible to add an empirical correction to the quantity found in the solution, thus shortening the process very considerably. A. G. L.

Ammonium Persulphate as a Substitute for Lead Peroxide in the Colorimetric Estimation of Manganese. Harry E. Walters. (*Proc. Engineers' Soc., Western Pennsylvania*, xvii., 257.)—In the following method the manganese is oxidized by means of ammonium persulphate in the presence of a silver salt: Samples of 0·2 gramme each of the test-steel and of a steel of known percentage of manganese are placed in test-tubes, and 10 c.c. of nitric acid (specific gravity 1·20) added to each. The tubes are heated in a water-bath till solution is complete and nitrous fumes are driven off. Fifteen c.c. of a solution of silver nitrate containing 0·02 gramme of $AgNO_3$ are added, and then immediately 1 gramme of solid ammonium persulphate, and the tubes again warmed, when oxidation will commence. After another half-minute's heating the tubes are placed in a cold-water bath. After oxidation is over and the tubes are cool, they are compared as usual.

Pig-irons and slags are treated in the same way as steels, except that slags should be moistened before adding the acid, and the solution filtered from suspended matter. In this case, the 15 c.c. of silver nitrate solution are used to wash the filter-paper. In dissolving shot or chilled iron samples, a little persulphate may be added to aid the solution of the sample.

The advantages claimed for the method are that it does away with the decantation of the solution from the excess of lead dioxide, that less acid is used, and that the oxidation is effected more rapidly at a lower temperature. The results quoted show that the two methods furnish practically identical results.

In experimenting with the method, it was found that solutions of ammonium persulphate could not be used instead of the solid, and also that if the salt was perfectly dry the reaction was incomplete. The author makes it a practice to add 10 c.c. water to each 1 lb. bottle of the dry salt a day or two before use.

A. G. L.

Detection of Small Quantities of Nickel in the Presence of Cobalt. H. Ditz. (*Zeits. f. angew. Chem.*, 1901, 894.)—Advantage is taken of the property of tartaric acid to prevent the precipitation of cobalt; but, instead of precipitating with hydrogen sulphide, as in Villiers' process (ANALYST, 1900, 305), the nickel is precipitated as chromate.

The neutral solution is placed in a capacious flask, and potassium chromate is added in slight excess. It is heated nearly to boiling, and 5 to 10 grammes of Rochelle salt are added. After this is all dissolved, the liquid is boiled vigorously for several minutes; then it is allowed to cool and diluted with water, if it be coloured very intensely. Any cobalt chromate precipitated is thus brought into solution again; but if nickel be present, a brown precipitate settles to the bottom of the flask, where it can be readily detected in the green liquid, even though the amount of nickel be very small. The precipitate contains a small proportion of cobalt. A. M.

The Quantitative Separation and Determination of Uranium. Edward F. Kern. (*Journ. Amer. Chem. Soc.*, xxiii., 685.)—As the result of his investigation, the author finds that the separation of uranium from iron, nickel, cobalt, zinc, and manganese is best effected by means of either a saturated solution of sodium carbonate, in which the uranium is completely soluble on prolonged boiling, or else by extracting the iron with ether and separating uranium from the other metals by means of a saturated solution of ammonium carbonate. In this method, which is to be preferred to the first, the solution of the mixed chlorides in hydrochloric acid (of specific gravity 1·10) is extracted three times with ether free from alcohol, which has previously been shaken with hydrochloric acid of the same density. From the alkalies and alkaline earths, uranium may be separated by electrolysis in the presence of sodium acetate and acetic acid; by repeated precipitation with ammonia in hot solutions containing ammonium chloride; or by precipitation with ammonium phosphate in the presence of ammonium acetate, the precipitate in this case being boiled for at least fifteen minutes before filtering. The pyrophosphate should be heated to low redness in a porcelain crucible; should reduction occur, it may be remedied by moistening with strong nitric acid and reigniting.

The author also finds that the reduction of U_3O_8 to UO_2 is unreliable as a check, and that the most rapid method of determining uranium consists in reducing the sulphate solution to $U(SO_4)_2$ with zinc, aluminium, or magnesium, and titrating with potassium permanganate. In hydrochloric acid solution, or with stannous chloride as a reducing agent, the reduction is carried beyond UCl_4, sometimes reaching UCl_3, and the results are consequently utterly unreliable. The preceding methods were tried practically in estimating uranium in pitchblende, those based on the ether separation giving the best results. A. G. L.

The Precipitation and Separation of Silver Electrolytically. W. H. Fulweiler and Edgar F. Smith. (*Journ. Amer Chem Soc.*, xxiii., 582.)—A series of experiments was made in which silver was deposited electrolytically from cyanide solutions (2 to 5 grammes KCN per 125 c.c.) containing, in addition to the silver, copper, copper and cadmium, copper, cadmium and zinc, and copper, cadmium, zinc, and nickel. The time required to deposit 0·1 gramme of silver at a temperature of 55° to 80° C. was three to six hours, using a current of about 0·02 ampères for a surface of 100 square centimetres, the voltage being 1·2. The results obtained were satisfactory; but if both cadmium and zinc are present, it appears to be essential to heat the liquid to 75° or 80° C. before passing the current to prevent the precipitation of some of the cadmium, and the presence of nickel slightly retards the deposition of the silver. A. G. L.

The Electrolytic Separation of Mercury from Copper. **C. Roscoe Spare
nd Edgar F. Smith.** (*Journ. Amer. Chem. Soc.*, xxiii., 579.)—In reply to some
riticisms on this method the authors give results of a series of experiments in
hich mercury was separated from copper electrolytically in potassium cyanide
lution (2·to 6 grammes KCN per 125 c.c.), the quantity of copper present being
om one-third to eleven times the weight of the mercury. The current used was
1 to 0·6 ampères for a surface of 125 square centimetres, the voltage being 1·1 to
7, the temperature of the liquid 60° to 65° C., and the time required to deposit
12 gramme of mercury, two to five hours. All the results obtained were satisfactory,
d the deposit was always perfectly free from copper. Some experiments in which
ercury was deposited from solutions containing copper and cadmium, and copper,
dmium and zinc, also gave good results. A. G. L.

The Electrolytic Method applied to Uranium. **L. Gavit Kollock and Edgar
. Smith.** (*Journ. Amer. Chem. Soc.*, xxiii., 607.)—The authors obtained good
esults by electrolyzing solutions of uranium acetate containing a little free acetic
cid, as well as solutions of uranium sulphate and nitrate. The uranium is deposited
s hydrated protosesquioxide, ignited and weighed as U_3O_8. The dilution used was
25 c.c. for 0·1 to 0·2 gramme U_3O_8, the temperature 65° to 75°, and the duration of
ach experiment four to seven hours. The current employed for 107 square centi-
netres of cathode surface was 0·05 to 0·55 ampères and 4 to 16 volts in the case of
he acetate solutions, and 0·02 to 0·04 ampères and 2 to 4 volts for the sulphate and
itrate solutions. Good results were also obtained in separating uranium from
arium, calcium, magnesium, and zinc in solutions of the acetates, provided that
vaporation of the liquid was prevented as far as possible, as otherwise the uranium
ydrate tends to enclose the separated salts, which cannot then be washed out.
his method, however, cannot be used to separate uranium from iron, nickel, or
obalt. A. G. L.

The Electrolytic Determination of Molybdenum. **L. Gavit Kollock and
dgar F. Smith.** (*Journ. Amer. Chem. Soc.*, xxiii., 669.)—The authors found that
o deposition took place on passing a current through neutral solutions of sodium
olybdate heated to 75° C. On adding 2 drops of concentrated sulphuric acid,
owever, the liquid turned dark blue, and then, as this colour gradually disappeared,
he cathode became coated with a black deposit of the hydrated sesquioxide.
oncordant results could not be obtained by merely washing and drying this
eposit, but very satisfactory results were found by dissolving the deposit in dilute
itric acid, evaporating the solution to dryness, and heating the residue of molybdic
cid. The electrolyte contained 0·1 to 0·25 gramme MoO_3 in 125 c.c.; the current
sed was 0·02 to 0·04 ampère and 2 volts for a cathode surface of 107 square centi-
etres; and the time required for the deposition was two to four hours.

It was also found that, instead of acidulating the solutions with sulphuric acid,
cetic acid (1 c.c. of 29 per cent.) could be used; the time required in this case is
ather longer, and it is best to add the acetic acid gradually. Since the presence

of sodium acetate was found not to interfere with the electrolysis, the method was applied to the analysis of molybdenite as follows: The mineral was fused with sodium carbonate and nitrate, the melt dissolved in water, the solution filtered from insoluble oxides, and the filtrate electrolyzed after acidulating with acetic acid and boiling off the carbonic acid. In the liquid left after the electrolysis, the sulphur was estimated as barium sulphate. The results obtained were fairly satisfactory.

A. G. L.

On the Solubility of certain Metallic Oxides in Solutions of Sodium or Ammonium Salicylate. J. Wolff. (*Zeit. anal. Chem.*, 1901, xl., 459-462.)—Concentrated solutions of sodium or ammonium salicylates possess the power of dissolving freshly-precipitated metallic oxides, the amount dissolved depending on the nature of the oxide. Copper hydroxide is exceedingly soluble even in the cold. Iron hydroxide and aluminium hydroxide, however, do not dissolve until the temperature reaches 80° C., and are both much less soluble than copper hydroxide.

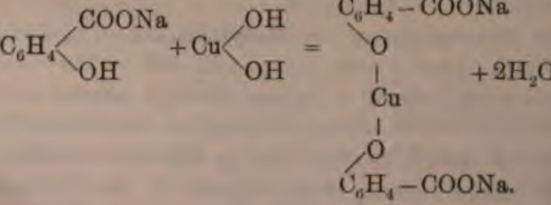
On adding a slight excess of sodium hydroxide to the solutions in sodium salicylate, the red iron solution becomes lighter in colour, but there is no change in the copper or aluminium solutions. In the presence of a large excess of sodium hydroxide the iron solution gives a precipitate of ferric hydroxide, the copper solution changes from emerald green to bright blue.

A slight excess of ammonium hydroxide does not change the appearance of any of the ammonium salicylate solutions, whilst a large excess of ammonia produces a blue coloration in the copper solution, but does not give any precipitate with either the iron or aluminium solutions.

Hydrogen sulphide completely precipitated the iron as sulphide.

The solubility of copper hydroxide in concentrated sodium salicylate solution depends upon the formation of copper-sodium salicylate, which the author has obtained in a crystalline condition. A solution of this salt is reducible by glucose.

The author explains the formation of this compound by the following equation:



$$2C_6H_4\begin{cases}COONa\\OH\end{cases} + Cu\begin{cases}OH\\OH\end{cases} = \begin{array}{l}C_6H_4-COONa\\ \diagdown O\\ \quad|\\ \quad Cu\\ \quad|\\ \diagup O\\ C_6H_4-COONa.\end{array} + 2H_2O.$$

C. A. M.

Estimation of Chlorates in Electrolytic Bleach and Chlorate Liquors. H. Ditz. (*Chem. Zeit.*, 1901, xxv., 727.)—The process which the author has already described (ANALYST, 1900, xxv., 80) for the iodometric analysis of mixtures of chlorates and hypochlorites has been reinvestigated and modified slightly to suit the purposes indicated in the title. The apparatus remains as before, but the main bottle should hold 1·5 litres, and the washing flask, which is now a cylinder, about

20 c.c. It has been found that the one hour's standing is not necessary, the reaction being finished well within five minutes. The volume of strong hydrochloric acid must not be reduced below 50 c.c., and 500 or 600 c.c. of diluting water should be employed in order to hinder any action between that acid and the potassium iodide. The method now runs as follows: If a chlorate liquor is to be analysed, a volume containing about 0·1 gramme of $KClO_3$ should be taken; if a determination of chlorate in mixtures of chlorates and hypochlorites, the volume should be such that between 40 and 50 c.c. of decinormal thiosulphate shall be required in the final titration. If, in the latter case, 25 c.c. or less of the liquor are sufficient, 50 c.c. of strong hydrochloric acid are employed; if the liquor is weaker and more must be taken (*e.g.*, 50 c.c.), 100 c.c. of acid and a correspondingly larger quantity of diluting water are used. The liquor is brought into the flask with 10 c.c. of 10 per cent. potassium bromide solution, and after the washing tube has been two-thirds filled with 5 per cent. potassium iodide, the 50 c.c. of strong HCl are run in. The stopper of the washing tube is immediately inserted, and the apparatus allowed to rest for five minutes. Then 500 or 600 c.c. of water are introduced and 20 c.c. of 5 per cent. potassium iodide. After thorough shaking, the contents of the side tube are blown and rinsed back into the flask, and the liquid is titrated with thiosulphate. Hypochlorites must be determined separately, and their proportion deducted. Commercial chlorate liquors can be run hot into the flask as soon as the free chlorine has been boiled off, and the whole process can be finished in half an hour. It is quicker than, and quite as accurate as, the ferrous sulphate method, which latter cannot be used if much hypochlorite is present, and in which the reagent needs standardizing every day.

F. H. L.

Estimation of Phosphoric Acid in Soil by the Götz Process. E. Gully. (*Chem. Zeit.*, 1901, xxv., 419.)—By making certain modifications in this process, the author has rendered it simpler in operation, capable of dealing with larger quantities of phosphoric acid, and more suitable for the analysis of soils. The vessel in which the mechanical agitation is conducted is about 17 centimetres long, holding 65 to 70 c.c. The narrow part holds altogether 0·4 c.c., is 40 to 45 millimetres long, and is graduated into eighty equal divisions, two of which contain 5 cubic millimetres, and hold ammonium phospho-molybdate equivalent to about 0·23 milligramme of P_2O_5. The whole graduated tube thus holds a precipitate equal to 18·45 milligrammes of P_2O_5; but as Von Jüptner has already shown in the case of iron analysis, the yellow precipitate does not consolidate itself evenly in the tube, which must be calibrated with mercury, and checked on a large number of blank experiments with pure disodium phosphate. (Gully appends to his paper a table of the readings of a tube in which 80 divisions are equal to 18·43 milligrammes of P_2O_5, whereas 10, 20, and 40 are equal to 2·22, 4·42, and 9·10 milligrammes respectively.) In carrying out an analysis of soil, a quantity of the sample (from 2 to 10 grammes), which contains less P_2O_5 than the maximum capacity of the tube, is brought into acid solution, and the silica removed. Hydrochloric acid is driven off by repeated evaporations with strong nitric acid, the dry residue is taken up in 1·19 nitric acid, and the solution is diluted to 50 c.c. with the same liquid. The graduated portion of the agitation tube is filled with 25 per cent. ammonium nitrate solution, carefully avoiding air bubbles, 25 c.c. of the soil solution are pipetted in, another 10 c.c. of ammonium nitrate are introduced, and the whole is placed for at least ten minutes in a water-bath at 65° C. It is next taken out, 25 c.c. of Finkener's* molybdate solution are added, and thoroughly agitated (by hand) for one minute, watching that the liquid in the graduated portion of the tube mixes with the rest. The tube is then filled up with the ammonium nitrate solution, and put back in the water-bath, where it should remain immersed up to its neck for twenty minutes. It is next taken out, adjusted in the centrifugal machine, and revolved for four minutes at a speed of 1,100 to 1,200 revolutions per minute. The volume occupied by the precipitate is finally read off and calculated into P_2O_5.

If it is preferred to use less than 25 c.c. of the soil solution for precipitation, that quantity of fluid must be made up by adding the requisite amount of nitric acid. It is necessary that the nitric acid should have the specific gravity specified, and that the prescribed volume of it should be present; in all other respects, notably the temperature of the water-bath, adherence to the details given above is essential. To show the accuracy of the process, Gully gives a table of sixty-five moorland soils containing from 0·03 to 0·33 per cent. of P_2O_5 when analysed by the ordinary gravimetric method, the measurement figures for which show maximum differences of $+0{\cdot}009$ and $-0{\cdot}01$ per cent.; and he points out that, when the discrepancy is larger, the fault is probably due to the gravimetric process, which contains greater possibilities of error than the modified Götz method. F. H. L.

Determination of Phosphoric Acid as Phospho-Molybdic Anhydride. **A. Seyda.** (*Chem. Zeit.*, 1901, xxv., 759-768.)—The investigations described with great minuteness in this lengthy article fall into three divisions, including attempts to simplify the Meineke-Woy process (ANALYST, 1897, xxii., 250 and 333) for determining phosphoric acid with molybdenum only, a study of the effect of adding citric acid to the molybdate reagent in order to obtain a precipitate free from uncombined molybdic acid, and the elaboration of a method of converting the Wagner-Stutzer combined molybdenum-magnesium process into one in which the magnesian precipitation is avoided. The results of the various experiments prove: (1) In the determination of phosphoric acid as ammonium phospho-molybdate, the only source of error is a contamination of the precipitate with free molybdic acid—Woy's idea that "alkali" may be present being quite incorrect. (2) Using excess of molybdate solution contamination of the first precipitate with molybdic acid cannot be avoided with absolute certainty—whence it follows that no mere agitation process can be considered universally applicable. (3) If the phosphoric acid is only precipitated once, such contamination is most likely to be prevented by mechanically agitating the mixed liquids for fifteen minutes at the atmospheric temperature, while an addition of 20 c.c. of 10 per cent. citric acid solution to the whole is advisable. In this case, if iron is present, a temperature of 20° C. must not be exceeded, and the liquid must be filtered fifteen minutes after the end of the agitation. In the absence of iron the temperature may be allowed to rise to 30° C. (4) The precipitate is best purified from molybdic acid by dissolving it in ammonia, adding 50 to 100 c.c. of "dilute molybdate" (see below) and reprecipitating hot with nitric acid. In certain circumstances the process of purification must be repeated several times; if so, the strengths and volumes of the reagents employed must be kept as specified. (5) The precipitate can be filtered hot, and washed with "washing liquid" at 60° or 80° C. The filtrate and washings hould be preserved for twenty-four hours to note any imperfect precipitation of phosphoric acid. (6) The precipitate should be rinsed down from the sides of the Gooch crucible as it creeps up with 95 per cent. alcohol. (7) The yellow precipitate s only to be considered as completely converted into phospho-molybdic anhydride when it exhibits a uniform black and crystalline appearance all through.

---

* One hundred and sixty grammes of ammonium molybdate dissolved in 795 c.c. of cold water, and 320 c.c. of 0·925 ammonia, poured into a mixture of 1,710 c.c. of 1·2 nitric acid with 1,205 c.c. of water, preventing any rise of temperature by constant cooling.

The reagents required for the "Wagner-Meineke-Woy" process are as follows: 1a) *Wagner-Stutzer Molybdate:* 150 grammes of ammonium molybdate are dissolved in about 600 c.c. of warm water in a 2-litre flask; when cool, 1 litre of 1·19 nitric acid is introduced, and to the clear mixture 400 grammes of solid ammonium nitrate are added in small portions at a time, shaking repeatedly. The liquid is diluted to the mark, kept for several days and then filtered. So prepared, separation of molybdic acid will not occur. (1*b*) *Citric Acid Molybdate:* 10 grammes of powdered citric acid are dissolved in 1 litre of the above and filtered after twenty-four hours. This reagent is much more permanent than the foregoing. (2) *Nitric Acid:* 25 per cent., specific gravity 1·15; stored in a bottle with a rubber cork bearing a 0 c.c. pipette. (3) *Ammonia:* 8 per cent., specific gravity 0·967; stored in a bottle with a 10 c.c. pipette. (4) *Dilute Molybdate:* a solution containing 0·1 per cent. f ammonium molybdate and 10 per cent. of ammonium nitrate, in a wash-bottle. 5) *Washing Liquid:* 5 per cent. ammonium nitrate and 1 per cent. nitric acid. 6) *Alcohol:* 95 per cent. in a wash-bottle.

The original solution in which phosphoric acid has to be determined is preipitated with the molybdate according to Wagner's directions, placing the beaker or twenty or thirty minutes on the water-bath and then letting it settle. [It would appear that 60° C. is the "critical temperature" for the deposition of molybdic acid, and the liquid should be kept below that point. The rule applies even when citric acid is present, as that reagent loses its inhibitory power at the same temperature.] When clear, the liquid can be either filtered at once, or safely left for hours, even vernight. The precipitate is collected on a paper, well washed with the washing iquid, and the bulk of it is rinsed into a wide flat beaker with the dilute molybdate olution, which is ready in a wash-bottle. The residue on the paper is then dissolved by allowing 10 c.c. of the ammonia to run over its surface, but the paper is preferably not perforated. Finally the filter is washed three times with cold dilute molybdate. The ammoniacal solution should be quite clear, and always is so, as if the molybdate reagent is properly prepared iron is never thrown down with the yellow precipitate. The beaker is covered with a glass, placed on gauze, heated till the liquid begins to boil, removed from the flame, and 20 c.c. of the nitric acid are added boiling hot. After two or three gentle agitations the vessel is stood on the water-bath till the solution is clear, and then set aside. It is ready for filtration in five or ten minutes.

Filtration is done in a Gooch crucible, the precipitate being collected and washed with hot washing liquid. Ignition is performed in a modified form of the apparatus described by Woy. F. H. L.

The Examination of Enamel and the Fusibility of Silicates. E. Kochs and F. Seyfert. (*Zeits. f. angew. Chem.*, 1901, 719.)—The authors give a rule for calculating from their composition a fusibility coefficient for the silicates used for enamelling iron. The ratio of the number of molecules of alumina to the number of molecules of flux is divided by the ratio of the number of molecules of silica to the number of molecules of alumina. Or, expressing the rule algebraically:

$$q = \frac{T^2}{173 \cdot 4} F K.$$

where q = function of fusibility.
T = per cent. Al_2O_3.
K = „ „ SiO_2.
F = sum of the figures obtained by dividing the percentage of the other (flux) constituents each by its molecular weight.

The higher the function q is, the higher is the melting-point of the enamel. With the aid of this formula it is possible to calculate the addition or additions which must be made to raise or lower the function to any required extent. Examples are given of a number of actual determinations and experiments. A. M.

The Behaviour of Borax when Distilled with Methyl Alcohol. E. Polenske. (*Arb. Kaiserl. Gesundheit*, 1900, xvii., 564-568; through *Zeit. für Untersuchung. der Nahr. und Genussmittel*, 1901, iv., 801.)—When borax is distilled with methyl alcohol, 57 to 59 per cent. of the boric acid contained in the borax passes over in the distillate; about 50 per cent. comes over readily, the remainder slowly. From the methyl alcohol remaining in the distillation-flask, sodium metaborate, $NaBO_2 + 5CH_3OH$, crystallizes out. It was found that on fusion 1 molecule of borax would drive out the carbon dioxide from 3 molecules of sodium carbonate. The melt obtained by fusing together 1 molecule of borax and 1 molecule of sodium carbonate, and which had the composition of sodium metaborate, when distilled with methyl alcohol, gave a considerable quantity of boric acid in the distillate. Distillates from the melts containing more sodium carbonate were free from boric acid. When the different melts were dissolved in methyl alcohol, the substance $NaBO_2 + 5CH_3OH$ always separated out. Sodium metaborate, on distillation with methyl alcohol, gives off boric acid until the residue has the composition $Na_{10}B_8O_{17} (= 5Na_2O + 4B_2O_3)$. This residue must be considered as a mixture of sodium metaborate and soda. The latter prevents the formation of boric esters and the possibility of distillation. W. P. S.

Determination of Boric Acid. H. Lührig. (*Pharm. Centralh.*, 1901, xlii., 50-56; through *Zeit. für Untersuch. Nahr. und Genussmittel*, 1901, iv., 801, 802.)—

he author confirms the accuracy of Jörgensen's method for the estimation of boric cid (see also the ANALYST, 1900, xxv., 101, 102). By combining Gladding's method vith that of Jörgensen's, less accurate results were obtained than with the latter lone.

W. P. S.

The Quantitative Determination of Ozone. A. Ladenburg and R. Quasig. (*Ber. deutsch. chem. Ges.*, xxxiv., 7, 1184.)—It was recently pointed out by Laden- urg (*Berichte*, xxxiv., 631) that the present methods for the estimation of ozone uffer from the want of a check, and are consequently unreliable. It was also shown *loc. cit.*) that by direct weighing of the ozone, even when mixed with oxygen, such a heck could readily be obtained. Evidently this method itself might be used for the stimation of ozone, but it is not generally applicable, as it demands certain condi- ions which cannot always be fulfilled in practice. By its help, however, it is now ossible to determine the reliability of the various titrimetric methods in common use, nd the examination of the potassium iodide method forms the subject of the present aper. The authors reserve to themselves the right to examine other methods in the ame way later on.

In the potassium iodide method it is usual to lead the gas through the aqueous olution of the salt acidified with an equivalent amount of sulphuric or acetic acid, and itrate the liberated iodine with sodium thiosulphate solution. Proceeding in this ay, the authors always obtained results about 50 per cent. too high, and consequently dopted the method in which the ozone is led through a neutral solution, which is nly acidified just before the titration. This method gives very accurate results and as the advantage that there is no return of the blue colour, which always occurs in e first method after the lapse of several hours, and necessitates the addition of a rther considerable amount of sodium thiosulphate. This result is quite contrary that found by Brunck (*Berichte*, xxxiii., 1835) and to the current opinion. The gh results obtained by the first method may be due to a catalytic action of the zone on the oxygen and the solution of potassium iodide, or else to the formation of ydrogen peroxide during the reaction according to the equation—

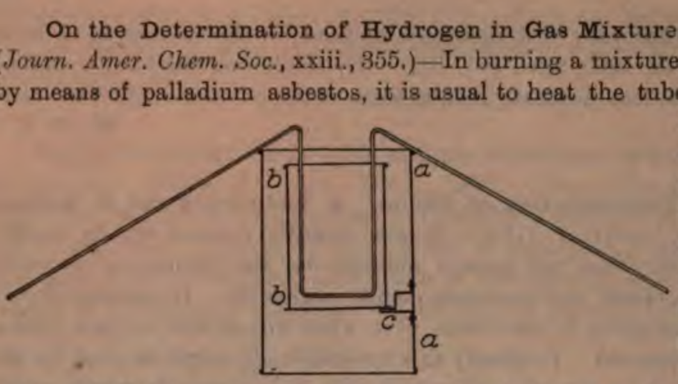
$$4O_3 + 10HI + H_2O = 5I_2 + H_2O_2 + 5H_2O + 3O_2,$$

hich represents the quantities found almost quantitatively.

The actual determinations were carried out as follows: Dry oxygen was passed rough a glass bulb of a capacity of nearly ½ litre, provided with two stopcocks, til the weight of the closed bulb remained constant. Ozone was then passed rough for about twenty minutes, and the bulb again weighed. The difference tween the two weighings, multiplied by 3, gives the weight of ozone present. The lb was then connected to two wash-bottles containing potassium iodide solution, d the ozone slowly sucked through them by means of a pump. When a neutral lution of potassium iodide was employed, only a trace of iodine was found in the cond wash-bottle. An equivalent quantity of sulphuric acid was then added, and e liberated iodine titrated in the usual way.

A. G. L.

On the Determination of Hydrogen in Gas Mixtures. Francis C. Phillips. (*Journ. Amer. Chem. Soc.*, xxiii., 355.)—In burning a mixture of hydrogen and oxygen by means of palladium asbestos, it is usual to heat the tube containing the asbestos in order to prevent condensation of the water formed. If a naked flame is used for this purpose there is danger of burning hydrocarbons with the hydrogen. In the form of apparatus shown, the heating is conveniently effected by means of boiling water contained in the cup-shaped vessel *b*, *b*, 18 centimetres deep and 12 centimetres wide, which rests on three supports riveted to the inside of the brass cylinder *a*, *a*, open at both ends, 30 centimetres long and 18 centimetres wide, the rims being strengthened by wires. One of the supports is shown at *c*. The cup is silvered on the outside and the cylinder on both sides. The polished silver surfaces serve to prevent radiation of the heat to the neighbouring gas-measuring apparatus. The cup is heated by a small flame placed underneath. The tube containing the asbestos has a total length of 70 centimetres and an internal diameter of 3 millimetres. A. G. L.



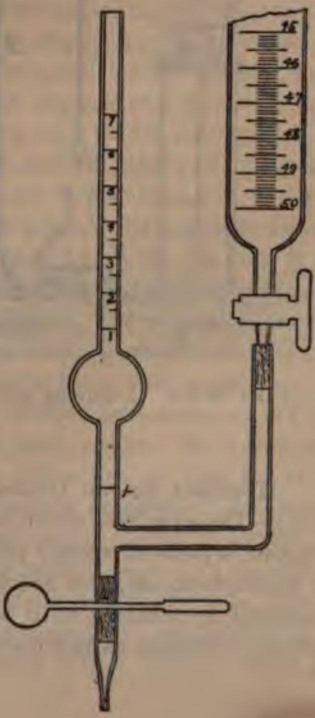
Method for Preparing Strictly Tenth-Normal, Fifth-Normal, etc., Hydrochloric or Nitric Acid. Richard R. Meade. (*Journ. Amer. Chem. Soc.*, xxiii., 343.) —The author has applied his method of making standard sulphuric acid (*Journ. Amer. Chem. Soc.*, xxiii., 12) to the preparation of standard hydrochloric and nitric acid. To prepare decinormal hydrochloric acid, 12·487 grammes pure crystallized copper sulphate are dissolved in 500 c.c. of water, the copper is precipitated electrolytically, and the solution transferred to a 1-litre graduated flask. A solution containing exactly 12·215 grammes crystallized barium chloride is then added, and the solution made up to the mark; after adding 2·6 c.c. of water to make up for the volume occupied by the precipitate, the whole is mixed, allowed to settle, and the clear liquid siphoned off and filtered through a dry filter.

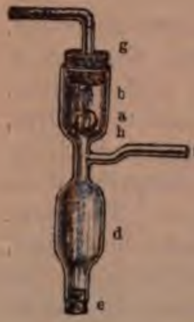
To prepare decinormal nitric acid, 13·076 grammes barium nitrate are substituted for the barium chloride. Solutions prepared in this way were found to be of exact strength and free from barium, but contained small quantities of sulphuric acid (up to 0·0320 gramme per litre). For many purposes it might be better to add a small excess of barium salt to avoid this. (*Cf.* Analyst, xxvi., 51.) A. G. L.

Methods of Standardizing Acid Solutions. Cyril G. Hopkins. (*Journ. Amer. Chem. Soc.*, xxiii., 727.)—The object of the investigation was to find the most exact method of standardizing solutions of hydrochloric or sulphuric acid. In the silver chloride method used, the hydrochloric acid was precipitated by a very slight excess only of silver nitrate, the precipitate filtered on a Gooch crucible and dried at 130° to 150° C. In the ammonium sulphate method excess of ammonia was added sulphuric acid, the solution evaporated, and the residue dried at 120° C. Both nethods gave excellent results. For the standardization by means of sodium, ter was weighed out under petroleum in a weighing-bottle, dissolved in , and, after diluting the solution with water, titrated with the acid solutions, phenolphthalein as indicator. The results agreed well amongst themselves, re higher than those obtained by the first two methods, owing, probably, to ties in the sodium. Incidentally they provided a means of comparing these with each other, and showed them to be in excellent agreement. The borax l, in which air-dried crystallized borax is titrated with the hydrochloric acid, limethyl orange as indicator, gave values which showed that the borax lost on continued exposure to the air. This was verified by exposing the salt to l weighing from time to time. Consequently the method is not reliable. obtained by electrolyzing solutions containing known weights of copper e, titrating the acid liberated with sodium hydrate solution, and using this dardize the hydrochloric acid, agreed well amongst themselves, but still were rly so good as those obtained by the first two methods, the maximum varia- the results found in the copper sulphate method being 0·048 milligrammes 1 c.c. solution, in the silver chloride method 0·002 milligramme HCl, and ammonium sulphate method 0·004 milligramme H_2SO_4 in 1 c.c. solution ively. Consequently the author concludes that these two methods are by far st suitable for standardizing acid solutions. A. G. L.

APPARATUS.

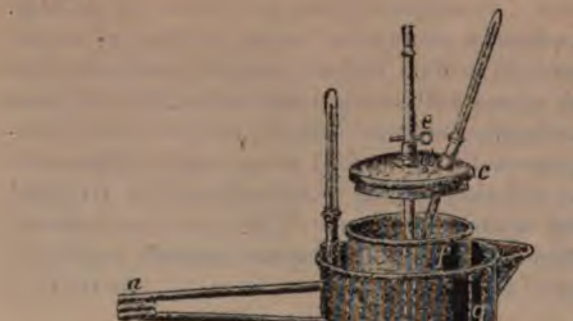
a Modified Form of the Ostwald Burette- tor. Allerton S. Cushman. (*Journ. Amer. Soc.*, xxiii., 484.)—In this calibrator the pipette scale engraved on its stem, as shown in the The pipette must have a capacity of 2 c.c. e mark A to about the middle of the scale. ibration is made starting with the burette filled zero mark, and the pipette filled to A. The c. of the burette are delivered into the pipette, ding is noted, and the pipette emptied to A; 2 c.c. are then introduced, and the operation ted down the scale. A second series of read- y be taken, starting with the burette filled to c. mark. Using this instrument, not a single g is necessary if the object is only to ascertain r the graduations of a given burette are suffi- uniform or not, and if absolute corrections are l it can be standardized much more easily e ordinary form. A. G. L.





Non-return Valve for Water-Pumps. L. Wacker. (*Chem. Zeit.*, 1901, xxv., 589.)—As shown by the diagram, this apparatus consists of a glass or metal ball-valve riding on a rubber seat inside a metal or glass casing. When the pump is working properly, the valve is lifted off its seat by the suction; but should any water return, the ball falls and retains the liquid in the upper chamber, whence it eventually drops into the lower cylinder, to be withdrawn by the removal of the cork at the bottom.

F. H. L.



The Engler-Ragosine Viscosimeter. A. Ragosine. (*Chem. Zeit.*, 1901, xxv., 628.)—The author has modified the Engler viscosimeter in one or two particulars, so as to render that instrument more convenient for determinations of viscosity at somewhat elevated temperatures. As shown by the illustration, the oil-container of the apparatus is provided with a tightly-fitting cover, 5 to 8 millimetres thick, and composed of a material which conducts heat badly. At *d* it has a 1 millimetre opening to admit air into the cup. The central rod is drilled with a transverse hole, through which a pin *e* is passed to hold the rod up while oil is running out; *a* is a circulating coil of copper tube, and *b* a draw-off pipe for the water. By this arrangement the temperature of the oil will not fall more than 0·1 or 0·3° C. during a determination of viscosity at 50° C.

F. H. L.

Modified Gooch Crucible. W. C. Heraeus. (*Zeits. f. angew. Chem.*, 1901, 922.)—The crucible, which was devised by Neubauer, has, instead of a perforated bottom, a piece of spongy platinum, so that no asbestos is required. Consequently, it is simple to use, and absolutely constant in weight. The precipitate is afterwards removed by treating with solvents. The modified crucible must not be used for barium sulphate, as this cannot be dissolved out of the pores of the platinum.

A. M.

REVIEWS.

Public Water Supplies. F. E. Turneaure and H. L. Russell. New York: John Wiley and Sons. Price 5 dollars.

This book is primarily concerned with the engineering side of the question of public water supplies, but it necessarily includes a consideration of the quality of the water obtained and supplied, and this involves a chapter on the criteria provided by chemical and bacteriological methods for determining the purity of a given source. It is evident that, as the whole section touching on this topic occupies less than 100 pages in a book of more than 700, nothing elaborate or exhaustive is to be expected. But what there is is sound, plain, and sensible, and, though not likely to be new or useful to the water analyst, will serve to guide the water engineer aright in the comprehension and interpretation of the analyst's results. In this respect the work is commendable. B. B.

A Manual of Determinative Bacteriology. By Frederick D. Chester. New York: The Macmillan Company. 1901. Pp. 401. Price $2·60.

This is not a text-book on bacteriology, but a work intended to be used as an analytical key to the numerous recognised races of bacteria, which has been elaborated by the author with the praiseworthy intention of lessening the difficulties which attend the identification of these organisms. The situation with regard to the successful accomplishment of the task upon which the author has ventured is one of special difficulty, for not only is the number of recognised forms of bacteria to be dealt with very large and their descriptions widely scattered, but, far more difficult than this, the nature of many so-called "species" of bacteria is too variable, or ill-defined, to admit of exact description, and consequently of accurate identification. It is possible to overcome the first-mentioned difficulty in a satisfactory manner, and the author has accomplished this part of his task at the expense of much time and labour, with the happy result that his book contains a very useful description of some eight hundred organisms. But regarding the ill-defined nature of many so-called species of bacteria, it is impossible for any method of identification to be a *complete* success, because we have not at our disposal at present the requisite knowledge to differentiate accurately numbers of different forms of bacteria—in fact, it is, perhaps, questionable how far we can accurately use the term "species" in connection with any of the recognised bacteria. For instance, the two well-known organisms *B. coli communis* and *B. typhosus* are types possessing marked characteristics by which they may be readily identified, but between these two types there is a chain of races connecting these two ideal species, whose characteristics are so indefinite that they cannot with certainty be classed with either. It is not the fault of the book, therefore, if it does not solve such difficulties as this, for it is limited by our present want of knowledge. But, as the author proposes in difficulties such as the one mentioned, it is possible even now to arrange a system of groups in which all doubtful organisms may be placed, even if they cannot be further identified at present. We hold that the science of bacteriology with regard to the questions of species and of means for their determination must be considered as occupying a tentative position, and if the book under notice is viewed in this light we have no hesitation in recommending it as a most useful work. We have tested it ourselves, and believe that if it is used carefully it fulfils its object of assisting in the determination of bacteria, so far as they can be determined, in a very successful manner. But here we should like to add the caution that it is essential that those who use this book should be well prepared by previous training in bacteriological methods, for bacteriology resembles all the other experimental sciences in demanding experience from its followers.

In general arrangement the book consists of two short chapters, introducing the author's systematic list of bacteria, which occupies the rest of the volume. The first chapter treats briefly on the morphology of the bacteria, and requires no special remark; but the second chapter, which deals mainly with conditions of growth, is of special interest, for in it the author has laid down the standard conditions under which bacteria should be grown, in order to study their characteristics in a systematic manner.

The author's list of bacteria is classified according to the order proposed by Migula. It may, perhaps, be satisfactory from a botanical point of view, but we do not consider the author was well advised to follow closely any botanical classification, in view of the general purpose of his book, which is to assist in determining species and not in classifying them. There is a danger under such circumstances that simplicity in determination may have to be sacrificed at times to the less important purposes of classification, and we are inclined to think that this is shown with regard to the organisms classified by the author under the genus *Mycobacterium*. These organisms, as a genus, are difficult to differentiate from the genus *Bacterium*, and we think if they had been united under one head it would have simplified and improved the author's system of determination. It is desirable, also, to call attention to another point in the book which is confusing to the reader. The manner in which the author's classified list of bacteria is introduced on page 55 is very badly chosen. This list is the main feature of the book, and occupies more than three hundred of its pages, but it commences in the middle of a page, without any special heading to call attention to its importance. These faults, to which we draw attention, and others we have noticed, are, however, of minor importance, and do not seriously impair the real value of the book, which we confidently recommend to all who have to overcome the difficulties of identifying unknown bacteria; but, as we have attempted to show in this review, the book must not be expected to act as a guide beyond the limits of our imperfect knowledge concerning the specific distinctions of these micro-organisms, and it can only be used with advantage by those who are skilled in bacteriological methods. A. J. B.

EXAMINATION UNDER THE BUREAU OF MINES AMENDMENT ACT OF BRITISH COLUMBIA, 1899.

The half-yearly examination for the qualification of Provincial Assayer, which was held at Victoria, British Columbia, on November 19, 1901, and the four subsequent days, resulted in the following gentlemen being granted licenses: D. A. Ayres, Trail; N. Colinson, Victoria; W. Stone Marshall, Duncans, and G. H. Comrie, Vancouver.

The examiners were: W. F. Robertson, provincial mineralogist, Chairman; T. Rhymer Marshall, D.Sc. (Edin.), of Glasgow; J. Cuthbert Welch, F.C.S., Chief Chemist Canadian Smelting Works, Trail; and H. Carmichael, provincial assayer, Secretary.

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VOLUME XXVII.—No. 311.
FEBRUARY, 1902.

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THE ANALYST.

FEBRUARY, 1902.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

HE annual general meeting of the Society was held on Wednesday evening, anuary 22, 1902, in the Chemical Society's Rooms, Burlington House. The President Dr. J. Augustus Voelcker) occupied the chair.

The President having read the notice convening the meeting, the minutes of the revious annual general meeting, held on February 5, 1901, were read and confirmed.

Messrs. B. Kitto and D. Lloyd Howard were appointed scrutators of the ballot-apers for the election of Officers and Council for 1902.

The Hon. Treasurer (Mr. E. W. Voelcker) presented his annual report for 1901.

Dr. Rideal moved that the Hon. Treasurer's report should be adopted, and that vote of thanks should be accorded to the Hon. Treasurer for his services during the ast year.

The motion was seconded by Dr. Schidrowitz, and carried unanimously.

The Hon. Treasurer, having responded, proposed a vote of thanks to the uditors (Messrs. L. Kidgell Boseley and Martin Priest), which was seconded by Ir. John White, and unanimously passed.

Mr. Hehner proposed a vote of thanks to the President and Council of the hemical Society for their kindness in having continued to allow the Society of ublic Analysts to use their rooms at Burlington House for meeting purposes during he past year. Dr. Dyer seconded the proposition, which was carried unanimously.

The President then delivered his annual address.

Dr. Dyer proposed a vote of thanks to the President for his address, coupled vith a request that he would allow the address to be printed in the Society's roceedings. The proposition was seconded by Mr. H. Droop Richmond, and was arried unanimously.

The President having responded, a vote of thanks to the Hon. Secretaries for heir services during the past year was proposed by Mr. Jenkins, seconded by Ir. Kitto, and carried unanimously.

Mr. Bevan (Hon. Secretary) having acknowledged the vote of thanks,

The President announced that the scrutators had reported that the Officers and ouncil for 1902 had been duly elected, in accordance with the nominations made by he Council in December, 1901.

Mr. Bevan then read the following list of Officers and Council elected for 1902:

President.—J. Augustus Voelcker, M.A., B.Sc., Ph.D.

Past-Presidents.—M. A. Adams, F.R.C.S., Alfred H. Allen, Sir Charles A. Cameron, C.B., M.D., F.R.C.S., A. Dupré, Ph.D., F.R.S., Bernard Dyer, D.Sc., W. W. Fisher, M.A., Otto Hehner, Alfred Hill, M.D., J. Muter, Ph.D., Thos. Stevenson, M.D., F.R.C.P.

Vice-Presidents.—Wm. Chattaway, Henry Leffmann, M.A., M.D., Francis Sutton.

Hon. Treasurer.—E. W. Voelcker, A.R.S.M.

Hon. Secretaries.—Edward J. Bevan, Alfred C. Chapman.

Other Members of Council.—Julian L. Baker, Bertram Blount, Lawrence Briant, W. J. Dibdin, Arthur E. Ekins, Thomas Fairley, R. H. Harland, James Hendrick, B.Sc., Julius Lewkowitsch, M.A., Ph.D., W. F. Lowe, Clarence A. Seyler, B.Sc., W. Collingwood Williams, B.Sc.

The proceedings then terminated.

The monthly meeting of the Society was held in the Chemical Society's Rooms on Wednesday evening, January 22, immediately following the annual general meeting. The President (Dr. J. Augustus Voelcker) occupied the chair.

The minutes of the previous monthly meeting were read and confirmed.

A certificate of proposal for election to membership in favour of Dr. L. T. Thorne, F.I.C., was read for the second time; and a certificate in favour of Dr. J. J. L. van Rijn, Consulting and Analytical Chemist, Director of the Royal Agricultural Experimental Station, Maastricht, Holland, was read for the first time.

Messrs. Horatio Ballantyne, M. Hunter, M.A., E. A. Lewis, Frederick J. Lloyd, Albert E. Parkes, and Thomas Tickle, B.Sc., were elected members of the Society.

The following papers were read: "Note on Reichard's 'Silver' Method for the Determination of Morphine in Opium," by Philip Schidrowitz, Ph.D.; "Note on a Sample of Coffee containing Starch," by Cecil H. Cribb, B.Sc.; "Note on a Sample of Artificial Coffee Beans," by Cecil H. Cribb, B.Sc.; and a "Note on Spurious Cream of Tartar," by John White.

THE MANNITIC FERMENTATION OF WINE.

By Philip Schidrowitz, Ph.D.

(*Read at the Meeting, December* 11, 1901.)

Gayon and Dubourg (*Ann. de l'Inst. Pasteur*, xv., 527-569) have recently published a paper on the "Mannitol Ferment," describing the properties of this micro-organism, and more particularly the changes produced by it in various sugars and allied compounds.[*] The paper alluded to records the continuation of the work published by the same authors in 1894 (*loc. cit.*, viii., 109). In view of the renewed interest which has been aroused in regard to this subject, and also of the fact that the published analytical data in connection with the sickness caused in wines by the mannitol micro-organism is by no means of an exhaustive nature, I believe that it may be of interest to the Society to give a brief account of my experience in this direction, more especially as I have been able to make some observations which have not previously been noticed.

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[*] The final paragraph of an abstract of Gayon and Dubourg's paper which appeared in the *Chem. Centralblatt.* 1901, ii. [10], 648, reads as follows: "It is apparent from the description given of the mannitol ferment that it cannot be the cause of 'sicknesses' in wines." I give the translation (which is a perfectly accurate one) *from the German* as it appears in the *Journal of the Society of Chemical Industry*, 1901, p. 1010). I was so surprised at this statement that I consulted the original (French) paper, and found that what the authors actually say is: "The results which we have obtained in our work have confirmed our previous studies, inasmuch as they tend to prove that the mannitic ferment is distinct from the 'ferment de la tourne' and the bitterness causing ferment, and that *the changes produced*

The mannitic sickness of wine was practically unknown, or, rather, unrecognised, until 1899, although it is probable that, under favourable conditions, it has occurred since the days when wine was first manufactured. The mannitic disease was first noticed in Algerian wines; in the first instance in fig, and later in grape wines. P. Carles (*Compt. Rend.*, 1891, cxii., 811) believed that the presence of mannitol in Algerian grape-wines was due to their having been adulterated with fig-wine, which latter usually contains a considerable quantity (0·6 to 0·8 per cent.) of this substance; but subsequent investigations by Jégou (*Journ. Pharm. Chim.* [5], 1893, xxviii., 103) and Lebanneur (*Répert. Pharm.*, 1893, xlix., 10) and others proved that it undoubtedly occurred in genuine Algerian wines. Very shortly afterwards (1892) the fact that the mannitic disease may also, under certain conditions, attack French wines, was demonstrated in a forcible and unpleasant manner.

The mannitic fermentation of wine is caused by a distinctive bacillus. For a description of this organism, its habits and properties, reference must be made to the researches of Gayon and Dubourg (*loc. cit.*), but it may be said here that Roos (*Journ. de Pharm. et de Chim.*, xxvii., 405) apparently first noticed that the conversion of saccharine matter into mannitol could be brought about by micro-organisms found in mannitic wines. Gayon and Dubourg (1894, *loc. cit.*) subsequently isolated the specific mannitol bacterium, which, morphologically, forms very short rodlets, showing a pronounced tendency to grow together in small packets. They also described the morphological and other differences between this ferment and those causing other wine-sicknesses (bitterness, "tourne," etc.). In their latest paper the chemists named have proved that of all the sugars and allied substances (pentoses, alcohols) lævulose alone is capable of being converted into mannitol. This conversion is accompanied by the formation of glycerin, fixed and volatile acids (chiefly lactic and acetic acids), and carbonic acid, but no ethylic alcohol is produced. Glucose and other sugars are converted into alcohol, acids, glycerin, etc., the proportion of fixed and volatile acids, however, being considerably higher than is the case in normal alcoholic fermentations. The *Bacillus gummosus* of Happ (*Dissert.*, Basle, 1893), which produces "slimy" fermentations, and, *inter alia*, mannitol, appears to be distinct from the micro-organism of Gayon and Dubourg, and this applies also to the bacteria described by Laborde (*Ann. de la Brass.*, 1898, 337).

In wine the disease develops during the primary fermentation, and the conditions favourable, or, rather, necessary, for its appearance, are a high temperature

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by it in wines are different from those produced by the two other sicknesses" (the italics are mine). It appears to be desirable to call attention to this error in translation somewhat prominently, as it might be the cause of considerable misapprehension, and might possibly have serious results.

and a lack of acidity in the grapes. The optimum temperature for the formation of mannitol from lævulose (Gayon and Dubourg, 1901, *loc. cit.*) is 35° C., and this figure is in accordance with practical experience, which has shown that if the temperature of the fermenting vessels is kept under 35° to 37° there is little danger of mannitic infection. The practical results of mannitic fermentation in wine are that a part of the lævulose of the must becomes converted into mannitol instead of into ethylic alcohol, and this is accompanied with the formation of an excessive amount of acids (principally lactic and acetic). The wine acquires an unpleasant sour-sweet taste, remains persistently turbid, and, if the malady acquires a firm hold and is not checked in good time, it is rendered quite unsaleable.

The quantity of mannitol that will form naturally in a solution of lævulose under favourable conditions is (according to Gayon and Dubourg), roughly, 70 grammes per litre. If the acid produced be neutralized with chalk, as much as 200 grammes per litre may be obtained. In wine the maximum observed is about 30 grammes per litre. Gayon and Dubourg (394, *loc. cit.*) describe an Algerian wine with 31·46 and a Spanish wine with 23·50 grammes per litre, but the highest figure for a French wine (red) that I have been able to find is 12·36 grammes per litre. The figure relating to wine No. 2 (see table)—namely, 10·26 grammes per litre—is, to the best of my knowledge, the next highest on record for a claret. Mannitol, if present in moderate quantities, is of itself not harmful from a commercial point of view.* It becomes a danger, however, if it is associated with any considerable quantity of unfermented sugar, with an excess of volatile acid and a large number of bacteria. Thus wines Nos. 6 to 8 all contained a fair amount of mannitol, but very little sugar or volatile acid; they obviously had been sick, but recovered, and at the time of analysis were quite sound. On the other hand, Nos. 3 to 5 contained little mannitol, but the high sugar and volatile acid in these samples were danger-signals which were corroborated by the microscopical examination and general physical condition of the wines. The chief danger (that is, in wines which are not already hopeless, such as 1 and 2) is the presence of unfermented sugar, as this will probably lead to a continuation of mannitic fermentation, together with its attendant evils. It is, however, not certain that the mischief will stop when the whole of the sugar has fermented, for Péglion (*Centralblatt f. Bakter.* [2], iv., 73) states that under certain conditions the mannitic bacteria will convert alcohol into acetic acid; but this certainly requires further confirmation. In any case it is advisable to make a microscopical examination of all suspected samples, and this

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* It has hitherto been universally held that the mannitol found in wine undergoes no further change, and remains present as such; but in this connection it may be well to remember that a number of organisms are known which produce either a modified alcoholic fermentation in mannitol, convert it into mannose, or reconvert it into lævulose. It seems conceivable that such organisms *might* be present in a mannitic wine, and produce ulterior changes in the same. The organisms that may be mentioned in this connection are: (*a*) *B. ethaceticus* and *B. ethaceto-succinicus* (Frankland, *Ann. de l'Inst. Pasteur*, 1892, 653; Frankland and Frew, *Journ. Chem. Soc.*, 1892, 254, and Frankland and Lumsden, *loc. cit.*, 132). The latter converts mannitol into alcohol, formic acid, acetic acid, and succinic acid, and the former has much the same action except that no succinic acid is produced by it; (*b*) *B. xylinum*, which (*Deutsche Essig Industrie*, 1899), according to Hager, converts mannitol into lævulose; (*c*) Bertram's sorbitose bacillus, which has the same action as *B. xylinum* (Vincent and Delachanal, *Compt. Rend.*, cxxv., 716); and (*d*) a micro-organism described by Péré (*Ann. de l'Inst. Pasteur*, 1898, xii., 63), which under certain conditions converts mannitol into mannose.

			sis.		H_2SO_4).	$CH_3.COOH$).				Tartar.				
Group A.—Wines completely spoilt ...	1	1895	1896	84·3	5·70	0·33	40·89	38·66	3·55	—	3·23	6·30	1·0014	6·22
	2	1895	1896	85·0	6·47	0·43	43·59	42·01	3·33	—	2·58	6·59	1·0027	10·26
[*Group A'*].—Wines of same growths as above, but of normal (non-mannitic) vintages	[1']	1896	1897	82·8	3·49	—	24·64	24·28	2·66	1·66	1·35	7·42	0·9958	—
	[2']	1900	1901	93·1	3·19	—	28·64	27·37	2·72	1·52	2 27	8·23	0·9960	—
Group B.—Wines rendered inferior and dangerous. Query whether saleable ...	3	1899	1900	87·3	4·07	1·70	34·60	25·88	2·60	2·14	6·72	7·80	0·9993	1·15
	4	1899	1900	92·0	3·75	1·26	29·85	26·72	2·36	2·01	4·13	8·15	0·9964	1·89
	5	1899	1900	91·3	3·88	1·43	31·18	26·39	2·25	2·34	5·79	6·81	0·9972	1·54
[*Group B'*].—Wines of same growths as B, but of normal (non-mannitic) vintages...	[3']	1897	1898	86·3	3·44	—	28·77	27·98	2·79	1·06	2·79	7·56	0·9969	—
	[4']	1900	1901	106·8	3·36	—	29 17	26·05	2·21	1·44	4·12	7·98	0·9950	—
Group C.—Wines containing a considerable quantity of mannitol. Had been sick, but recovered, and were sound when analysed	6	1895	1896	87·9	4·06	0·23	25·95	25·60	2·74	—	1·34	—	0·9971	2·59
	7	1895	1896	85·7	3 50	0·14	22·06	21·94	2·37	—	1·11	—	0·9945	3·52
	8	1895	1896	103·1	3·89	—	30·79	30·70	3·03	—	1·09	—	0·9959	1·57
Group D.—Old wines of exceptionally fine vintages containing mannitol	9	1865	1896	90·7	4·17	—	26·82	26·72	2·18	2·38	1·11	7·99	0·9957	0·66
	10	1875	1896	89·3	4·19	—	25·33	—	2·58	—	nil ?	5·16	0·9955	0·50

Note.—**Results are expressed in grammes per litre.**

as a rule furnishes sufficient indication as to the likelihood of any further bacterial mischief.

In view of the researches of Gayon and Dubourg (*loc. cit.*), of Carles (*loc. cit.*, and *Feuille Viniale de la Gironde*, 1895, and 1896, x., 12), of Jégou, of Lebanneur (*loc. cit.*), of Roos (*Journ. de Pharm. et de Chim.* [5], xxvii., 405, and *Mémoires de la Soc. Phys. et Natur. de Bordeaux*, July 28, 1892, iv., 3), there is little doubt that under ordinary circumstances the mannitic fermentation of wine produces not only a very high total acidity, but also an excess of volatile acid, the latter forming, roughly, one-third to one-half of the whole. That there are exceptions to this rule, however, is evidenced by the figures relating to wines Nos. 1 and 2, in which, although the total acid is high, the volatile acid is not above the normal. I have not been able to find any other cases of this kind recorded in the literature on the subject. On the other hand, in Nos. 3 to 5 the figures for the total acid are not particularly high, and the volatile acid in each case is excessive.

Analytical Methods.—The best *qualitative* test for mannitol in wine is undoubtedly that recommended by Gayon and Dubourg (*loc. cit.*), which consists in allowing a few c.c. of the wine to evaporate on a watch-glass at a low temperature, and, after a time (twenty-four hours as a rule suffice), examining the residue under the microscope. If mannitol is present (1 gramme per litre and upwards) extremely characteristic fine silky needles will be observed. It is best, however, to decolorize the wine first by means of basic lead acetate solution, or, better still, by shaking with a little powdered yellow oxide of mercury. *Quantitative* methods have been devised by Jégou (*Journ. Pharm. Chim.* [5], 1893, xxviii., 103), by Carles (*Compt. Rend.*, 1891, cxii., 811), and by Gayon and Dubourg (*loc. cit.*). Of these the first is, in my opinion, rather too complicated, and the second not very accurate. Gayon and Dubourg's process is fairly simple, gives good results, and, as far as my experience goes, is certainly preferable to the others. In my laboratory it is carried out as follows: 50 c.c. of the wine are evaporated on the water-bath to a syrup (adding 2-3 grammes of sand), and then allowed to stand for two or three days in a cool place. At the end of this time the mannitol will have crystallized out. One hundred c.c. of 85 per cent. alcohol, saturated at the room temperature with mannitol, are then gradually added, stirring vigorously meanwhile, and the whole mass then transferred to a filter, on which it is allowed to drain for two hours. Thereupon the filter is extracted in a soxhlet for one hour with 100 c.c. of 85 per cent. alcohol, four-fifths of the alcohol are distilled off, a little animal charcoal is added to decolorize, the whole is filtered, and then washed twice with about 50 c.c. of hot 85 per cent. alcohol. The filtrate is evaporated at 60°, and the residue, which is practically pure mannitol, weighed as such. If any considerable quantity of sugar is present in the wine it is best to ferment this out before estimating the mannitol. From a fairly large number of mannitic wines examined by the author, the analyses referring to some characteristic samples have been selected, and are given in the table. They are arranged into four groups—A, B, C, D—and for the sake of comparison analyses of wines from the same chateaux, but of normal vintage, are added to groups A and B. The headings to the table sufficiently explain the nature of the wines, but I may add that they were all clarets, and, with the exception of Nos. 6 to 8, of well-known growths.

Nos. 6 to 8 were also wines from the Gironde, but of unknown growth. Nos. 9 and 10 represent two wines which must be classed amongst the finest clarets of the last fifty years.

With regard to Group A, I have already alluded to the remarkable lack of volatile acid, and the comparatively low sugar, but, in addition, I may draw attention to the specific gravity, which is above 1·000—a most unusual thing, but readily explained by the extremely high extract which, in its turn, is of course due to the large proportion of mannitol. These wines were analysed some ten or eleven months after the vintage, and were then in a hopeless condition. It seems likely that if they had been examined immediately after the main fermentation they might have been saved. With regard to the wines in Group B, they were undoubtedly mannitic, notwithstanding the very moderate percentage of mannitol. The figures appear to lend point to the question raised above (see note, p. 44) concerning the possibility of a transformation of mannitol into ulterior products. These wines might have been safeguarded against any further formation of mannitol by pasteurization, but, in view of the high sugar and volatile acid, it is doubtful whether, at the best, they would be saleable or capable of standing bottling. The wines in Group C, although containing from 1·5 to 3·5 grammes per litre of mannitol, were quite sound, and it is evident that they had been attacked by the mannitol bacteria, but, either as a result of careful handling (cooling during fermentation, pasteurizing, and so on), or some fortuitous circumstance, such as a fall of temperature, or a particularly vigorous yeast, they had recovered.

To guard against the danger of mannitic fermentation, the use of overripe grapes—which lack acid—should be avoided when possible, but if this is inexpedient it may be practicable to raise the acidity of the must by judicious blending, or by an addition of 1 to 3 grammes of tartaric acid per litre of must, which is, in my opinion, both legitimate and likely to have the desired effect. Further, appliances for attemperating the *cuve* during hot seasons should be at hand whenever there is an adequate water-supply, and if the expense is not beyond the means of the proprietor. Unfortunately these conditions are not always attainable. If the wine is not quite hopeless after the main fermentation is well over, immediate pasteurization will prove an efficient safeguard in most cases, but the operation must (when the wine is very young) be accompanied by a subsequent treatment with a culture yeast, otherwise the wine will not develop.

DISCUSSION.

Mr. CHAPMAN said that in all the earlier statements it was maintained that mannitol was formed in considerable quantities when organic liquids became ropy, and that some distinct connection existed between the two phenomena. Certainly in the case of sugar solutions which had become viscous very appreciable amounts of mannitol were formed, and there appeared to be some close relationship between the bacteria responsible for the two changes. That being so, he would like to ask Dr. Schidrowitz whether, so far as he knew, any such connection had been observed in the fermentation of wine; that is to say, whether wines in which mannitol had been formed to a considerable extent also showed, as a rule, any tendency to become ropy. Dr. Schidrowitz had stated on the authority of a good many Continental observers that high temperatures and lack of acidity were conditions favouring the mannitic fermentation, and there seemed to be very little doubt that the former certainly did constitute a predisposing cause. On the other hand, he (Mr. Chapman) had seen statements to the effect that in the fermentation of wine mannitol was frequently formed in quantity when the fermentation had experienced a serious check. The production of mannitol had also been stated by Peglion to be an anaerobic phenomenon, acetic acid being apparently formed in its place when the organism was grown under aerobic conditions.

Mr. **Henry Symons** (Totnes) said that he had often found difficulties to arise in the case of cider from a deficiency of acid. The acid was a natural preservative, and a certain proportion appeared to be necessary.

Dr. **Schidrowitz** thought it was settled that the mannitic bacilli which caused ropiness in sugar solutions were quite distinct from the mannitic bacillus of wine. The point was that the latter did not produce ropiness in wine. It was different from Laborde's bacillus; and from what could be gathered from the literature on the subject of Happ's bacillus, he had come to the conclusion that they were entirely different. He had never observed anything approaching ropiness in wine as the result of the action of the mannitic bacillus. He thought that the mannitic bacillus was not a facultative anaerobe, but an anaerobe proper, and that when surface action occurred it must be due to an entirely different bacillus.

ADDENDUM.

Analyst, xxvi., p. 264.—The results in Mr. Richmond's paper are expressed in parts per 100,000.

REPORT OF THE CONJOINT COMMITTEE ON THE DETECTION AND APPROXIMATE ESTIMATION OF MINUTE QUANTITIES OF ARSENIC IN BEER, BREWING MATERIALS, FOOD-STUFFS AND FUELS.

The joint committee of the Society of Chemical Industry, and of the Society of Public Analysts, appointed in March, 1901, and consisting of: Messrs. Otto Hehner (Chairman), Alfred H. Allen, Alfred C. Chapman, C. Estcourt, David Howard, Arthur R. Ling,* Drs. Rudolph Messel, and Leonard T. Thorne, report as follows:

After an examination of various methods, the committee recommend that of Marsh-Berzelius.

Materials Required.

Hydrochloric Acid.—The purest hydrochloric acid obtainable is very rarely free from arsenic. To the "pure" acid, as purchased for analysis, diluted with distilled water to a specific gravity of 1·10, sufficient bromine is added to colour it strongly yellow (about 5 c.c. per litre); sulphurous acid, either gaseous or in aqueous solution, is then added *in excess*, and the mixture is allowed to stand for at least twelve hours. Or hydrobromic acid and sulphurous acid may be used. The acid is then boiled till about one-fifth has evaporated, and the residue can either be used direct, or may be distilled, the whole of the arsenic having volatilized with the first portion.

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* Mr. Ling also acted as secretary.

Sulphuric Acid.—This is more frequently obtainable arsenic-free than hydrochloric acid. If not procurable, to about half a litre of sulphuric acid, "pure for analysis," a few grammes of sodium chloride are added and the mixture distilled from a non-tubulated glass retort, the first portion of about 50 c.c. being rejected. For the purpose of the test to be described, one volume of the distilled acid is diluted with four volumes of water.

Nitric acid can, as a rule, be obtained free from arsenic without much difficulty, the pure redistilled acid being used. This should be tested by evaporating 20 c.c. in a porcelain dish, which should then be washed out with dilute acid, and tested as described in this report.

The purified acids should be prepared as required, and should not be stored for any length of time. If this be unavoidable, however, Jena flasks are to be preferred, since most bottle glass is liable to communicate traces of arsenic.

Zinc.—Arsenic-free zinc is obtainable from chemical dealers. It should be regranulated by melting it and pouring it from some height into cold water.*

Lime.—Caustic lime, even when made from white marble, is not always free from arsenic. A selection must, therefore, be made from various samples. If pure lime is not obtainable magnesia may equally well be used, and can more readily be obtained of sufficient purity.

Calcium Chloride.—This salt often contains arsenic, and before being used as a drying agent must be freed from the volatilizable part of the impurity by moistening it with strong hydrochloric acid, fusing, and regranulating.

APPARATUS.

A bottle or flask, holding about 200 c.c. (for frothing materials preferably wider at top than bottom), is fitted with a doubly-bored cork, india-rubber stopper or with a ground-in glass connection, carrying a tapped funnel holding about 50 c.c. and an exit tube. The latter is connected with a drying tube containing, first, a roll of blotting paper soaked in lead acetate solution and dried, or a layer of cotton wool prepared in a similar way, then a wad of cotton-wool, then a layer of granulated calcium chloride, and finally a thick wad of cotton-wool. To this tube is fitted a hard glass tube, drawn out as shown in the figure, and of such external diameter that at the place where the arsenic-mirror is to be expected the tube just passes through a No. 13 Birmingham wire gauge (corresponding with 0·092 inch). The exact size is not material, but all tubes used for standards and tests should be as nearly as practicable of the same diameter. A good Bunsen flame is used to heat the hard glass tube close to the constriction. About one inch of tube, including the shoulder, ought to be red-hot.

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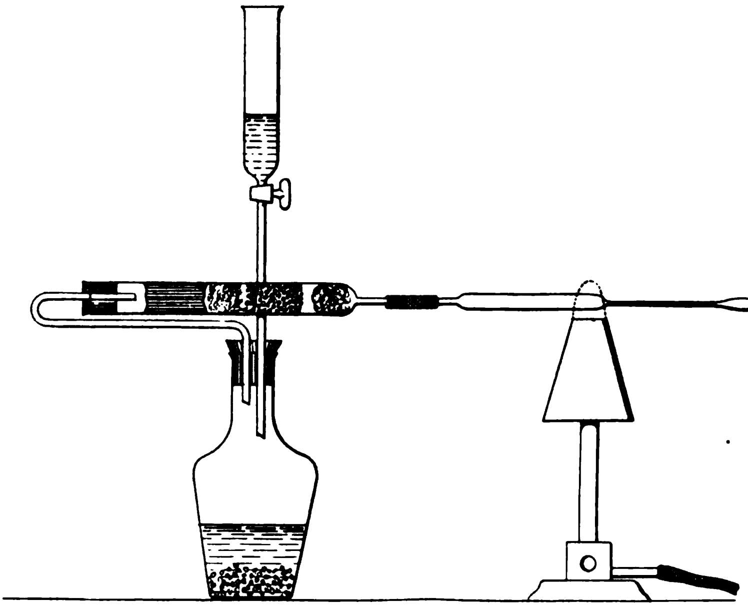
* Mr. A. H. Allen holds it to be essential, both for a regular evolution of hydrogen and for the formation of uniformly deposited brown-coloured mirrors, that the zinc should contain a trace of iron.

A piece of moderately fine copper gauze (about one inch square) wrapped round the portion of the tube to be heated assists in insuring an equal distribution of heat. A suitable form of apparatus is shown in the figure.



MODE OF TESTING.

About 20 grammes of zinc are placed in the bottle, and washed with water to clean the surface, as particles of dust may contain arsenic; all parts of the apparatus are connected, and a sufficient quantity of acid (prepared as previously described) allowed to flow from the funnel, so as to cause a fairly brisk evolution of hydrogen. When the hydrogen flame—which, during the heating of the tube should be kept at as uniform a height as possible (about a quarter of an inch)—burns with a round, not pointed tip, all air has been removed from the apparatus. The Bunsen burner should then be placed under the hard glass tube as described, and more acid (10 to 20 c.c. is generally enough) run in as required. With good materials no trace of a mirror is obtained within half an hour. Great care must be taken that when additions of acid are made to the zinc no bubble of air is introduced, since in presence of air the arsenic mirror may become black and unevenly distributed, whilst it is brown when the experiment has been properly conducted.

Should the blank experiment not be satisfactory it must be ascertained by changing the materials methodically whether the fault lies with the acid, zinc, other materials, or with the apparatus.

Preparation of Standard Mirrors.—When a satisfactory blank experiment has been obtained a series of standard mirrors must be prepared under the following conditions:

A hydrochloric acid solution of arsenious oxide, containing in each cubic centietre 0·001 milligramme As_4O_6, is prepared by diluting a stronger solution with istilled water. Two c.c. of this solution (equal to 0·002 milligramme of arsenious xide) are introduced into the apparatus, a new tube having been joined to the drying be. If the zinc is sensitive, a distinct brown mirror is obtained after twenty inutes. It is important to note that some "pure" zinc is, from a cause at present nknown, not sufficiently sensitive; that is to say, the addition of minute quantities arsenic produces no mirror. The portion of the tube containing the mirror should sealed off while still filled with hydrogen; in contact with air the mirrors gradually de. Mirrors are now similarly made with 0·004, 0·006, 0·008, and 0·01 milligramme arsenious oxide. With a little practice it is easy to obtain the deposits of arsenic eatly and equally distributed. The standard mirrors, properly marked, are mounted n a white card or porcelain slip. It is to be understood that the first stage of every st must be a blank of at least twenty minutes.

Hydrochloric acid is somewhat more sensitive than sulphuric acid—that is to y, it gives rather denser mirrors with minute quantities of arsenic. If for one eason or another sulphuric acid is preferred by the operator, he must make a set of andard mirrors with sulphuric acid, and use these for comparison.

Organic materials, such as beer, yeast, etc., cannot be tested, when sulphuric acid used, without destruction of the organic matter, whilst, as a rule, they can be irectly tested with hydrochloric acid. However, many materials are met with in hich it is preferable to destroy the organic matter.

Procedure without Destruction of Organic Matter.

The apparatus is started, and a blank experiment allowed to go on for twenty inutes. If no trace of a deposit is obtained, 10 c.c. of the liquid to be tested and bout 10 c.c. of hydrochloric acid are put into the funnel, and slowly introduced into e bottle without air-bubbles. Some materials (beers, for example) are apt to froth, ence the necessity for slow introduction. If after about ten minutes no mirror ppears, another 10 c.c. of the liquid, with 10 c.c. of hydrochloric acid, are added, nd the experiment continued for fifteen to twenty minutes, acid being from time to me added as may appear necessary.

Malt.—Fifty grammes of the malt are placed in a 300 c.c. separator funnel rnished with a stopcock; 50 c.c. of hydrochloric acid, prepared as described, and 0 c.c. of water are warmed to about 50° C., and poured on the malt. The whole then allowed to digest for fifteen to twenty minutes, with frequent agitation, nd the acid then allowed to run off by the stopcock. About 60 c.c. of the acid liquor thus obtained, of which every 20 c.c. contains the arsenic from 10 grammes of e malt.

Hops.—Twenty grammes of hops are digested with 100 c.c. of dilute hydroloric acid (1 volume of the purified acid to 1 volume of water) at about 50° C. for alf an hour, 50 c.c. of the strained-off liquid being used for the test.

Sugar and other *brewing materials* are dissolved in water, 10 c.c. of acid added, d the solution tested direct, operating upon from 10 to 20 grammes of material.

DESTRUCTION OF ORGANIC MATTER.

(*a*) *Acid Method.*—Ten grammes of the substance are placed in a 3½-inch porcelain crucible, and covered with pure redistilled nitric acid (about 10 to 15 c.c.). The whole is then heated on a sand-bath until the evolution of brown fumes ceases. Three c.c. of concentrated arsenic-free sulphuric acid are then added, and the heating continued till the mass just begins to char, when a further quantity of 5 c.c. of nitric acid is added. The heating is now continued till all the acid is expelled, leaving in the crucible a black, nearly dry, charred mass. The crucible is about half filled with water and a few c.c. of hydrochloric acid, or of dilute sulphuric acid, run in (according as the one or the other is to be used in the Marsh apparatus), the whole being allowed to extract for about half an hour on a water-bath. It is then filtered into a porcelain basin, the charred mass washed with hot water, and the filtrate concentrated down to about 30 c.c., which is allowed to cool, and is then ready for the test. It is essential that the mass should be *thoroughly* charred, and that the solution, when filtered, should be colourless.

In the case of *beer*, 10 to 20 c.c. are evaporated to dryness on a water-bath, and the residue oxidized as above stated.

Hops.—10 c.c. of pure nitric acid and 5 c.c. of pure concentrated sulphuric acid are mixed in a 3½-inch porcelain crucible, and the hops are then added in small portions at a time, each quantity being thoroughly disintegrated by pressure under the acid with a glass rod, a further quantity of 5 c.c. of nitric acid being added when about half the hops have been thus introduced. The crucible with its contents is then cautiously warmed so as to avoid frothing over. When the evolution of dense red fumes ceases the heating is increased, and the acids are evaporated on a sand-bath, and the dry charred mass extracted with dilute acid, filtered, concentrated, and introduced into the Marsh apparatus in the ordinary way. It may be noted that with many English hops of relatively fine texture the addition of the second quantity of nitric acid above recommended is unnecessary.

When, owing to the presence of larger quantities of arsenic, smaller amounts of substance—*e.g.*, 0·5 gramme to 2 grammes—are taken, the quantities of acids recommended above may, of course, be reduced.

(*b*) *Basic Method.*—The materials are mixed with pure lime or magnesia (1 gramme for 20 c.c. of beer), dried and incinerated. For sugars or other solid materials about half their weight of base is employed. The ash is dissolved in hydrochloric acid, and the solution tested. This method is not recommended for hops.

Of *coal* or other *fuel*, after careful sampling, two portions of 1 gramme each are weighed. One portion is incinerated in a platinum dish in a muffle, and the hydrochloric acid extract of the ash tested for "non-volatile arsenic." The other is intimately mixed with 1 gramme of lime or magnesia and also incinerated. The hydrochloric acid extract of the latter gives the "total arsenic," the difference between the two determinations being the "volatile arsenic." It may in some cases be found that the above-mentioned quantity of fuel gives a mirror too dense to be measured. When this is the case the hydrochloric acid extract is diluted to a determinate volume and an aliquot portion taken.

Sulphites.—The sulphurous acid must be oxidized by bromine, the excess of the latter being removed by heating.

The committee have convinced themselves that arsenic in both states of oxidation can be detected and estimated by the procedure described.

As an additional precaution a fresh tube should always be substituted for that containing the mirror, and the experiment continued for a further period of fifteen minutes. Should a second mirror be formed, the quantity of arsenic with which it corresponds is to be added to that shown by the first.

It must be understood that the tests are only approximate, and that mirrors corresponding with less than 0·003 milligramme of arsenious oxide in the quantity of materials taken cannot be safely relied upon. When a mirror has been obtained, a duplicate test should always be made to preclude error by accidental contamination.

The proof that the mirrors are arsenical is obtained as follows: The narrow portion of the tube containing the mirror (which should not be denser than that produced by 0·01 milligramme of arsenious oxide) is cut off, the hydrogen replaced by air, and the ends sealed up. The tube, held in the tongs, is then heated by drawing it repeatedly through the flame of a Bunsen lamp until the mirror has disappeared. On cooling, minute crystals of arsenious oxide deposit, the sparkling of which can be seen with the naked eye if the tube be held before a luminous flame, and which can be readily identified under the microscope by their crystalline form.

This test, as recommended, is one of such extreme delicacy, that with quantities of 20 grammes (or 20 c.c.) it will give an indication of the presence of 0·000015 per cent. (or one part in 7,000,000) of arsenious oxide. This would represent with solids $\frac{1}{1000}$ grain per pound, with liquids $\frac{1}{100}$ grain per gallon. It must be understood that the committee do not suggest any limits for traces of arsenic which may be regarded as negligible; but they desire to express the opinion that limits should officially be fixed by the Royal Commission or otherwise. This could be easily effected by prescribing the amounts of solids and liquids respectively to be taken for the test and the minimum mirror to be recognised.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

Detection of Powdered Cocoanut Shell in Ground Spices. A. L. Winton. (*Amer. Journ. Pharm.*, 1901, lxxiii., 552-555.)—According to the author, the adulteration of spices with powdered cocoanut shell is a very common practice in the United States.

The powdered shell contains the tissue elements of the mesocarp, endocarp, and outer testa, but the stone cells of the endocarp are the principal constituent. These cells can be recognised under the microscope by their porous, brownish-yellow cell-walls, which contain a dark brown substance, changing to reddish-brown on treatment with potassium hydroxide. They differ in one or more of these particulars from the stone cells of pepper, allspice, clove stems, walnut shells, almond shells, Brazil-nut shells, hazel-nut shells, peach stones, and olive stones.

Ferric chloride has no immediate effect upon the colour of the brown cells—a characteristic which distinguishes them from the cells of allspice, which are decolorized by potassium hydroxide and immediately changed to green by ferric chloride.

Spices adulterated with charred cocoanut shell show under the microscope black opaque fragments not bleached by *aqua regia* or nitric acid and potassium chlorate. Chemical analysis is also valuable, as is shown in the following table of results obtained by the author. The crude fibre obtained in the analysis is especially suitable for the microscopic detection of stone cells and other tissues.

	Black Pepper (Average of 14 Analyses).	Cloves (Average of 8 Analyses).	Allspice (Average of 3 Analyses).	Nutmeg (Average of 3 Analyses).	Cocoanut Shell (1 Analysis).
	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.
Water	11·96	7·81	9·78	3·63	7·36
Total ash	4·76	5·92	4·47	2·28	0·54
Ash, soluble in water ...	2·54	3·58	2·47	0·86	0·50
Ash, insoluble in HCl ...	0·47	0·06	0·03	0·00	0·00
Volatile ether extract ...	1·14	19·18	4·05	3·02	0·00
Non-volatile ether extract	8·42	6·49	5·84	36·70	0·25
Alcohol extract	9·62	14·87	11·97	10·77	1·12
Reducing substances by direct inversion calculated as starch ...	38·62	8·99	18·03	25·56	20·88
Starch by diastase method	34·15	2·74	3·04	23·72	0·73
Crude fibre	13·06	8·10	22·39	2·51	56·19
Total nitrogen	2·26	0·99	0·92	1·08	0·18
Oxygen absorbed by aqueous extract ...	—	2·33	1·24	—	0·23
Quercitannic acid equivalent to oxygen absorbed	—	18·19	9·71	—	1·82

C. A. M.

Sulphites as Food Preservatives. Lebbin and Kallman. (*Zeits. öffentl. Chem.*, 1901, vii., 324; through *Chem. Zeit. Rep.*, 1901, 293.)—From numerous experiments carried out on animals and on human beings, the authors have come to the conclusion that our present notions as to the toxicity of normal sulphites are wholly erroneous. With acid sulphites, however, the action is quite different, for most of them are as corrosive ("aggresiv") as free acids.

F. H. L.

Composition and Analysis of Absinthes. A. Hubert. (*Ann. de Chim. anal.*, 1901, vi., 409-413.)—Commercial absinthe or extract of absinthe is prepared by macerating certain plants in alcohol and distilling the extract, or by the addition of essential oils to the alcohol. The proportion of essential oils varies from 1·5 to 5 grammes per litre, and is such that the addition of an equal volume of water produces a characteristic opalescence.

A common adulteration is to replace the essential oils by various resins. For the estimation of the latter the author recommends the following method: 200 c.c. of the absinthe are distilled in a current of steam until the distillate passes over perfectly clear. The resinous residue is concentrated to a syrup and extracted with chloroform, and the extract evaporated. The residue should not exceed 0·5 gramme per litre.

The distillate is extracted several times with petroleum spirit (25 c.c. each time), the united extracts evaporated in a current of dry carbon dioxide, and the residue of essential oils weighed.

The colour of genuine absinthe, which should consist entirely of chlorophyll, is usually derived solely from one plant (*Artemisia pontica*), with the addition in some cases of hyssop. Inferior absinthes contain more or less colour derived from *artemisia*, but, in addition, other colouring principles such as that of veronica.

For the detection of foreign colouring matters, 20 c.c. of absinthe are shaken with several successive portions (5 c.c.) of chloroform until no more colour is extracted. The extract is evaporated and the residue taken up with distilled water. If the solution is colourless, or has only a faint yellow tint, the absinthe was free from artificial colours.

Three types of absinthes are sold, viz., those prepared with alcohol of 70 per cent., of 60 per cent., and of 50 per cent. strength. The following table gives the composition of different commercial samples:

Absinthes.	I.	II.	III.	IV.	V.	VI.	VII.	VIII.	IX.	X.	XI.	XII.
Specific gravity at 15° C.	0·9982	0·8966	0·9246	0·9340	0·9453	0·9353	0·9157					
Alcohol per cent. ...	48·0	67·6	55·0	50·0	44·0	50·0	59·0	49·0	47·0	55·0	65·5	57·0
Extract per litre ...	1·56	1·72	0·36	1·08	0·52	0·80	0·92	0·898	0·902	1·32	1·117	0·97
Reducing substances	trace	trace	—	trace								
Acidity	0·12	0·288	0·024	0·096	0·048	0·072	0·072	0·08	0·111	0·092	0·054	0·08
Aldehydes	0·126	0·155	0·005	0·025	0·091	0·10	0·052					
Furfural	0·0006	0·0004	0·0007	0·0002	0·0002	0·0003	0·0002					
Esters	0·035	0·071	0·005	0·123	0·070	0·070	0·070					
Essential oils ...	1·506	2·614	2·158	4·250	3·340	1·984	2·700	1·619	1·810	2·340	3·10	1·78

C. A. M.

The Detection of Benzoic Acid and Alkali Benzoates in Food. **J. de Brevans.** (*Journ. Pharm. Chim.*, 1901, xiv., 438-440.)—The aqueous extract of the substance under examination is filtered, acidified with a few drops of dilute sulphuric acid, and extracted with three successive portions (50 c.c.) of a mixture of equal parts of petroleum spirit and ethylic ether.

The united extracts are filtered and evaporated and the residue tested for saccharin (sweet taste) and salicylic acid. If benzoic acid be present it can be identified by its aromatic odour, irritating vapour on heating, and its crystalline form.

In the absence of salicylic acid or saccharin the aniline blue reaction is characteristic. About 0·5 c.c. of a solution of 0·02 gramme of rosaniline hydrochloride in 100 c.c. of aniline is boiled in a test-tube with a trace of the residue for about twenty minutes. If benzoic acid be present the liquid becomes violet-blue, and on adding a few drops of hydrochloric acid and shaking the tube a deep blue insoluble substance separates.

The colour obtained on adding a neutral solution of ferric chloride to benzoic acid, exactly neutralized with potassium hydroxide, furnishes a confirmatory test.

C. A. M.

Estimation of Alcohol in "Sulphuric" Ether. **F. Freyer.** (*Zeits. landw. Versuchsv. in Oesterr.*, 1901, iv., 955; through *Chem. Zeit. Rep.*, 1901, 308.)—This is a modification of Adam's process (ANALYST, 1899, xxiv., 259). It is first necessary to obtain an approximate idea of the amount of alcohol, or alcohol and water, in the ether to be analysed. Twenty c.c. of the sample are, therefore, agitated in a graduated tube with an equal volume of saturated calcium chloride solution. When the volume of residual ether is read off, the loss represents water and (or) alcohol. As 5 grammes of acetyl chloride, the quantity used in the analysis, are equivalent to 2·95 grammes of alcohol or 1·15 grammes of water, it is necessary that the amount of material weighed out for treatment shall not contain more than 1 gramme of liquid capable of being absorbed by calcium chloride solution, in case this should prove to be wholly water. So calculated, the proper quantity of the ether is weighed out in a 100 c.c. flask, and filled up to the mark with anhydrous ether. Twenty-five c.c. of this liquid are titrated as Adam described, but in standardizing the reagent 25 c.c. of the same pure ether should be present, as acetyl chloride may not be altogether without action upon it. Having made the determination, and knowing from the first experiment the amount of true ether present, the respective quantities of water and alcohol can be ascertained by an equation. If desirable, the treatment with acetyl chloride may be repeated on a larger amount of the sample, the maximum permissible quantity being calculated from the above figures, so as not to exceed the decomposing power of the 5 grammes of acetyl chloride taken. Expressed in another way, 1 gramme of ethyl alcohol is equivalent to 1·7 gramme of acetyl chloride, and 1 gramme of water to 4·33 grammes of the chloride.

F. H. L.

A Characteristic Reaction of Morphine. G. Fleury. (*Ann. de Chim. anal.*, 1901, vi., 417, 418.)—A solution of morphine in dilute sulphuric acid gives a faint rose coloration when shaken for six or eight minutes with a little lead dioxide (puce-coloured oxide), and the filtrate changes to deep brown on the addition of an excess of ammonium hydroxide. This coloration, which is due to the formation of protocatechuic acid, remains unchanged for several hours. Iodic acid can be used instead of lead dioxide, but is less satisfactory, since it yields a white precipitate of ammonium iodate.

C. A. M.

New Pharmaceutical Preparations. C. Kippenberger. (*Chem. Zeit.*, 1901, xxv., 1045.)—This article gives details as to the manufacture and clinical properties of a large number of proprietary drugs, etc.; the following abstract records merely the trade names and the true chemical composition of the substances mentioned:

Antiseptics and Disinfectants.—*Aniodol* and *aquinol* are bodies of imperfectly known constitution. The former is probably prepared from trioxymethylene, glycerol, and some allyl compound; the latter from formalin, glycerol, a potash soap, and thymol. *Guaiasanol* (*cf.* Analyst, 1901, xxvi., 159) is a hydrochloride, and crystallizes in white prisms, melting at 184° C. *Guaiamar* (Endemann) is guaiacol glyceryl ester. Kalle and Co. have introduced *triiodo*-m-*cresol*, made by the action of iodine upon a strongly alkaline solution of the phenol, and the similar *monoiodothymol* is now patented. Various objections having been made to *cinnamyl*-m-*cresol*, the same firm has replaced the latter constituent by one of its halogen derivatives. The corresponding "esters" of the chlorine and iodine derivatives of *m*-cresol have proved specially powerful antiseptics. F. Hofmann has patented the *ethyl ester of salicylcarboxylic acid*, to be used in the form of its water-soluble alkali-metal salts. Fehrlin has effected an improvement in the manufacture of *salol*, which he prepares by allowing phosphoryl chloride to act upon phenol sodium carbonate (or a corresponding alkaline-earth salt), followed by distillation in steam. G. Cohn has found that higher compounds can be obtained from salol by heating it with higher phenols, and the process is therapeutically useful because it enables monosalicylates of divalent phenols (*e.g.*, resorcinol) to be prepared more easily. *Resaldol* (Eichengrün) is a water-soluble acetyl compound obtained by the condensation of chlormethylsalicylic anhydride with resorcinol. It is stated to be toxic to bacteria not only in neutral, but also in alkaline menstrua. Eichengrün has also recommended the precisely similar condensation product from thymol. *Argent. thiohydrocarburosulphonicum*, or *ichthargan* (Analyst, *ut sup.*), is a brown amorphous, odourless powder containing 30 per cent. of silver, and soluble in water. Its concentrated solution is precipitated by common salt, also by albumin solution, which in large excess redissolves it. *Petrosulfol* is a substance chemically and physically identical with ichthyol. *Chininum lygosinatum*, prepared by the interaction of quinine salts and Fabinyi's sodium lygosinate, is used as an alcoholic solution for rendering dressings antiseptic. Boehringer and Sons have patented a method of making *alloxanphenols* by condensing alloxan and phenol in presence of hydrochloric or sulphuric acid, zinc chloride, etc.; these bodies are soluble in water, and free from taste. *Sodium* *biiodosalicylate* has been successfully introduced by Froloff as a substitute for iodoform; it is employed alone, or diluted with 50 to 97·5 per cent. of talc. Antiseptic, bactericidal, and air-resisting substances have been produced by combining hexamethylenetetramine, or its compounds, with tetriodopyrrol, or with an aromatic sulphonic acid, or halogen derivatives thereof, or with *p*-phenolsulphonic acid or naphtholsulphonic acid. Compounds of hexamethylenetetramine ethyl iodide with tetriodopyrrol, bromo- and chloro-pyrrol, have also been described. Phenols, such as naphthol combined with anthraquinone, phenanthrenequinone, and their derivatives have been introduced. Three modifications of *ibit* (ANALYST, *ut sup.*) are on the market; in these the second C_6H_2 nucleus is combined with (*a*) $(OH)_2.COOH$, (*b*) $O_2.BiI.COOH$, (*c*) $O_2.BiOH.COOH$ respectively. Ibit is claimed to be highly antiseptic, harmless, and capable of being sterilized. *Iodogallate* is airol, or airogen; *gallate* is dermatol; *naphthylate*, orphol; *methylenegallate*, bismal; *bromphenylate*, xeroform; but according to Duyk these all vary somewhat in composition (*cf.* Squire, 19th edition, 145). *Basol* is a substitute for lysol, and is a preparation of cresol-soap containing 50 per cent. of the phenol; it dissolves in water in all proportions to a clear liquid.

ANTIPYRETICS, ETC.—*Aspirin* is acetylsalicylic acid. Propionyl-, butyrl-, valeryl-, and higher condensation products of salicylic acid are now being proposed. A new benzyl ester of salicylic acid has been patented by the Act. Ges. für Anilin Fabrik., Berlin. It is a colourless and odourless oil, not easily soluble in ether or alcohol, readily hydrolyzed by alcoholic potassium hydroxide. Condensation products of antipyrine and primary aromatic amines (*e.g.*, aniline and *p*-toluidine) have been described. The former gives Knorr's pyrazolin reaction. Monoiodo-antipyrine has been introduced. *Thiopyrin* is thioantipyrine; it forms colourless crystals soluble in water and alcohol. *Eupyrin* (ANALYST, *loc. cit.*) forms pale greenish yellow, quite tasteless needles, having a faint odour of vanilla; it is easily soluble in alcohol, ether, and chloroform, not easily soluble in water; it melts at 87° to 88° C. Of the "*eosolsaure Salze*," the calcium, silver, and quinine compounds are now known. *Bromipin* (Squire, p. 153) has a specific gravity of 1·008 in 10 per cent. solution, of 1·331 in 33·3 per cent. solution, both at 20° C.; *iodipin* is used in 25 per cent. solution.

ANÆSTHETICS AND HYPNOTICS.—*New orthoform*, *m*-amido-*p*-oxybenzoic ester, and ordinary orthoform, *p*-amido-*m*-oxybenzoic ester, are rendered more powerful by being combined with chloral (Kalle and Co.), and the compounds are tasteless. *Chlorosonin* is a compound of chloral hydrate with hydroxylamine. *Nirvanin* is the methyl ester of diethylglycocoll-*p*-amido-*o*-oxybenzoic acid; it is proposed as a substitute for cocaine or orthoform, but its value is uncertain. "Compounds" made by mixing the hydrochloride or sulphate of morphine with borax or some other alkali salt, iodoform, and phenol, have been patented as local (dentists') anæsthetics. For *isomorphine* and *desoxymorphine*, *cf. Proc. Chem. Soc.*, 1900, xvi., 143. *Phosphorylquinine* (-chinin, Zimmer and Co.) is a tertiary quinine phosphate.

ANTITOXINS, ETC.—*Tuberculosamine tuberculate* and a compound of albumin with tuberculic acid are new preparations. *Antiethylin* is the active principle of a serum from horses treated regularly with alcohol; it is recommended for alcoholism.

lin is an analogous product for swine fever, etc. *Atrabilin* is a pale opalescent
rived from a glandula suprarenalis. *Brom-tannin-gelatin* is an American
of obvious composition; it is a grayish-yellow powder devoid of taste and
ontaining about 20 per cent. of Br; it is soluble in caustic alkalis, but almost
le in water and dilute acids. *Nucleol* is a pure nuclein obtained from yeast;
hitish powder soluble in warm water, but not in alcohol; it combines with
precipitated metallic oxides to yield *mercurol* (Hg), *nargol* (Ag), *cuprol* (Cu),
inol (Fe). This last contains 6 per cent. of iron; *ferratogen* is similar, but
s only 1 per cent. of Fe.

enose is a mixture of oatmeal with soluble acorn-malt extract. *Galactogen* is
ly soluble casein preparation; *roborat* is a food-stuff prepared from corn
LYST, *ut sup.*). *Usunify* is a labiate root found in the Soudan, and used
as a food. *Fersan* is a compound of iron, phosphorus, and albumin obtained
blood; *roborin* is prepared from ox or calf blood by cold sterilization
LYST, *ut sup.*).

NDRIES.—1. *Inorganic.*—The halogen compounds of arsenic are once again
sed in medicine. Dupoux has found the triiodide to be very irregular in
ition; sometimes it contains antimony, sometimes elemental arsenic, some-
ree iodine. Goldschmidt has described a method of preparing phosphoric
de by burning molten phosphorus on a wick. The burner is kept cool to
formation of amorphous phosphorus. Strontium bromide and iodide have
roduced as drugs.

Organic.—*Menthorol* is a mixture of *p*-chlorophenol and menthol, said to be
m the objectionable secondary action of the former alone. *Epicarnin* is a
ation product of cresotinic acid and *β*-naphthol. *Gasterin* is a preparation
e stomach-juices of dogs; Linossier has ascribed its utility to the unusual
of hydrochloric acid and pepsin it contains. Medicinal fatty oils treated
rbon dioxide are now being sold, cod-liver-oil in particular, which is said
uch easier of toleration, as it probably contains instable esters of its fatty

ring the past year the fourth edition of the German Pharmacopœia has been
d. Among the new preparations in it may be mentioned: A 99·6 to 99·7 per
ohol ("absolute"); an ether for anæsthetic work made from specially pure
ls; the hydrobromide of the alkaloid arecoline from areca-nut; *hydrargyrum*
um, the secondary mercuric salicylate; *coffeïno-natrium-salicylicum*, or
e double salt," a mixture of caffeine and sodium salicylate; *methylsulfonatum*,
ulphomethylethylmethane; *pyrazolonum phenyldimethylicum salicylicum*,
n, or antipyrine salicylate; *bismuthum subgallicum*; *barium chlorate*; *hydras-*
rochloride; and *gelatina alba*. Among severer methods of examination,
hydrate must be tested with sulphuric acid for foreign chlorine substitution
s; cocaine hydrochloride for isatropylcocaine; mercury, by complete
y in nitric acid, for antimony and tin; potassium nitrate for the sodium salt;
by its specific gravity, for medically useless phenols; morphine hydro-
for narcotine; while the essential oils must be more closely examined for
tive constituents.

F. H. L.

ORGANIC ANALYSIS.

Estimation of Alcohols or Phenols. A. Verley and F. Bölsing. (*Ber.*, 1901, xxxiv., 3354.)—Although alcohols or phenols only react slowly with organic acid anhydrides in the cold, presence of pyridine causes the reaction to be rapid and very often quantitative, according to the equation:

$$R.OH + (R'.CO)_2O + \text{pyridine} = R.O.CO.R' + R'COOH.\text{pyridine}.$$

An acid "mixture" is prepared of about 120 grammes of acetic anhydride and about 880 grammes of dry pyridine. If this is treated with water, pyridine acetate is formed at once; on adding an alkali it decomposes into alkali-metal acetate and free pyridine, and, as both of these bodies are neutral to phenolphthalein, the excess of acid can be titrated. In a 200 c.c. flask 1 or 2 grammes of the alcohol or phenol to be estimated are weighed, 25 c.c. of the mixture are added, and the whole is warmed for fifteen minutes on the water-bath without a condenser (no volatilization need be feared). After cooling 25 c.c. of water are run in, and the uncombined acid is titrated. Care must of course be taken that the mixture and the standard alkali are both at the same temperature. The process is specially suitable for the examination of essential oils, results obtained on a large number of them being quoted in the original.

F. H. L.

Detection and Estimation of Methyl Alcohol in Formalin. M. Duyk. (*Ann. de Chim. anal.*, 1901, vi., 407-409.)—The aldehyde is first converted into the non-volatile hexamethylene-tetramine by adding ammonium hydroxide drop by drop to 100 c.c. of the formaldehyde solution, previously diluted with the same quantity of water. After standing for several hours, the slightly alkaline liquid is mixed with a little sodium carbonate and distilled, and the distillate neutralized with sulphuric acid and redistilled. The fractions passing over between 65° and 100° C. are rectified and tested for methyl alcohol by adding red phosphorus and iodine, distilling and collecting the methyl iodide, and calculating its volume into the corresponding quantity of methyl alcohol.

C. A. M.

The Quantitative Estimation of Formaldehyde. L. Vanino and E. Seitter. (*Zeit. anal. Chem.*, 1901, xl., 587-589.)—In Smith's method of oxidizing the formaldehyde in an alkaline solution (ANALYST, xxi., 148), the end point of reaction is difficult to recognise.

The authors have found that the oxidation occurs rapidly and sharply by using the permanganate in a strong solution of sulphuric acid in the presence of hydrogen peroxide. The reactions which take place are shown in the following equations:

$$4KMnO_4 + 6H_2SO_4 + 5HCOH = 2K_2SO_4 + 4MnSO_4 + 5CO_2 + H_2O$$
$$(2KMnO_4 + 5H_2O + 3H_2SO_4 = K_2SO_4 + 2MnSO_4 + 8H_2O + 5O_2).$$

In making a determination, 35 c.c. of $\frac{N}{5}$ potassium permanganate solution are mixed with a mixture of 30 grammes of sulphuric acid and 50 c.c. of water previously cooled, and 5 c.c. of an approximately 1 per cent. solution of the formaldehyde added drop by drop, with continual agitation. The flask is now closed, and llowed to stand for ten minutes, an occasional shake being given. The excess of ermanganate is then titrated back with an approximately $\frac{N}{10}$ solution of hydrogen eroxide, previously standardized with potassium permanganate.

One c.c. of the permanganate solution corresponds to 0·001723 gramme of rmaldehyde.

A sample of formalin thus examined was found to contain, as the mean of three eterminations, 37·30 per cent. of formaldehyde, as against 37·08 per cent. by omijn's iodometric method (ANALYST, xxii., 221). C. A. M.

Olefines and Naphthenes in Petroleum. **L. Balbiano and V. Paolini.** *hem. Zeit.*, 1901, xxv., 932.)—Since naphthenes absorb bromine, Allen's process estimating olefines cannot be safely employed in the presence of the former. e authors have accordingly worked out a method based on Denigès' reaction for tiary alcohols (ANALYST, 1898, xxiii., 216). Their test for olefines is as follows: to 12 c.c. of a cold saturated solution of mercuric acetate are shaken for two or ree minutes with 3 or 4 c.c. of petroleum spirit. The tube is corked and set ide; if after twenty-four or thirty-six hours the aqueous liquid is rendered turbid th small white glittering lamellæ, olefines are present.

The test has been applied to three samples of Russian, American, and Italian etroleum with negative results, but a fourth sample, stated definitely to have been merican, contained an olefine in the fraction distilling under 100° C., which the uthors found to be hexylene. F. H. L.

The Proportion of Phenols in Essential Oils of Thyme. **Jeancard and atie.** (*Bull. Soc. Chim.*, 1901, xxv., 893-895.)—The amount of phenols is usually ade the basis of valuation of oil of thyme, and it is commonly accepted that a pure il should contain from 25 to 30 per cent. The authors, however, have met with ecimens of undoubted purity, in which the quantity of phenols varied from 5 to 60 per ent., and have therefore made experiments to determine the cause of this variation.

They find that on distilling the oil the fractions which pass over towards the nd contain the bulk of the phenols. They also find that the specific gravity of the il increases by about 0·0013 to 0·0015 with each increase of 1 per cent. of the roportion of phenols, and that the solubility in alcohol, the surface tension, and e viscosity, also increase with the amount of phenols.

They show that the same plant will yield products richer or poorer in phenols ccording to the manner in which the distillation is carried out, and conclude that e arbitrary standard of 25 to 30 per cent. might well be considerably raised.

C. A. M.

The Characteristics of Tomato Seed Oil. **L. Battaglia.** (*Ann. Soc. Chim. ilan*, 1901, 127; through *Ann. de Chim. anal.*, 1901, vi., 437.)—According to the uthor, tomato oil contains olein, linolin, stearin, myristin, and lecithin (2·3 per ent.). It has the following chemical and physical constants:

Specific gravity at 15° C.	0·922
Refractive index	1·473
Acid value	4·7 c.c. $\frac{N}{10}$ alkali.
Saponification value	190·4
Iodine value (Hübl)	106·9
Hehner value...	95·10
Reichert-Meissl value	18·93
Iodine value of fatty acids	112·0

C. A. M.

Candle Nut Oil. J. Lewkowitsch. (*Chem. Revue über d. Fett u. Harz-Ind.*, viii., 156.)—The constants obtained by the author for the oil of candle nuts (from *Aleurites moluccana*) are given below, together with those obtained by De Negri:

	De Negri.	Lewkowitsch.
Specific gravity	(at 15° C.) 0·920 to 0·926	(at 15·5° C.) 0·92565
Saponification value ...	184·0 „ 187·4	192·6
Hehner value	—	95·5
Iodine value	136·3 to 139·3	163·7
Refractive constant	(at 15° C.) 76·0 „ 75·5	(at 25° C.) 76·0 (at 20° C.) 78·5
Acetyl value	—	9·8
Hydroxy-acids (insoluble in petroleum ether)	—	0·21 per cent.

By extracting the peeled nuts 58·6 per cent. of oil was obtained. Commercially prepared cakes showed the following composition :

Oil	8·80 per cent.	
Moisture	10·00 „	
Ash	8·28 „	containing 23·52 per cent. K_2O. 53·04 „ P_2O_5.
Nitrogenous matter ...	46·16 „	
Cellulose	1·47 „	
Starch, sugar, etc. (by difference)	25·29 „	
	100·00 „	

A. G. L.

The Detection of Cane Sugar in Vegetable Substances by means of Invertase, and of Glucosides by means of Emulsin. E. Bourquelot. (*Journ. Pharm. Chim.*, 1901, xiv., 481-487.)—The detection of cane sugar by inversion with mineral acids is rendered uncertain by the fact that inulins, starches, and glucosides also undergo inversion.

This difficulty is obviated by the use of invertase, for although it also causes the inversion of raffinose and gentianose, these sugars can be distinguished by their optical properties from cane sugar.

The author obtains a preparation free from enzymes other than invertase by shaking a fresh top-fermentation yeast with 95 per cent. alcohol, allowing the mixture stand for thirty minutes, then draining off the liquid, and drying the residue idly at 30° C. One gramme of this product is triturated in 100 c.c. of water urated with thymol and filtered, yielding an active solution which will keep for out a week.

In using it in examining parts of plants, the latter are first treated with boiling ohol (for fifteen minutes under a reflux condenser) to destroy any soluble enzymes at may be present. The alcoholic extract is evaporated on the water-bath with e addition of some precipitated calcium carbonate, and the residue taken up with ter containing thymol and filtered. Ten c.c. of the filtered liquid are then mixed th 10 c.c. of the invertase extract, and a blank experiment made with 10 c.c. of the rate, mixed with 10 c.c. of water containing thymol, and left for three days at ° to 17° C. The amount of cane sugar can then be determined in the usual way by olarimetrical examination of the liquids before and after inversion.

After the three days the liquid is heated to 100° C. to destroy the invertase, and en cold treated with emulsin in the proportion of 0·05 gramme to 40 c.c., and ain left at the ordinary temperature. In the presence of glucosides inverted by ulsin, the formation of reducing sugar will be indicated by the polarimeter and tion on Fehling's solution. C. A. M.

Detection of Indican in Urine. C. Strzyzowski. (*Oesterr. Chem. Zeit.*, 1901. ., 465.)—Twenty c.c. of the urine are taken for examination. If the specific gravity ceeds 1·015, 10 c.c. of a 10 per cent. solution of normal lead acetate are added; if e specific gravity is 1·015, or less, 5 c.c. of the lead and 5 c.c. of water are used, us producing 30 c.c. altogether. The mixture is filtered through a dry paper, and c.c. of the clear filtrate are treated first with 1 drop of a 1 per cent. solution of tassium chlorate, then with 5 c.c. of chloroform, and finally with 15 c.c. of pure ming hydrochloric acid (specific gravity, 1·19), shaking repeatedly. In ten or teen minutes the maximum indigo-coloration is attained. Should the separating loroform be distinctly blue, a second, or even a third, drop of the chlorate lution may be used, shaking immediately; but in general 1 drop (0·5 milligramme $KClO_3$) is quite sufficient. F. H. L.

Estimation of Beta-Hydroxybutyric Acid in Urine. P. Bergell. (*Zeits. ysiol. Chem.*, 1901, xxxiii., 310; through *Chem. Zeit. Rep.*, 1901, 299.)—One hundred 300 c.c. of urine are made faintly alkaline with sodium carbonate, and concentrated the water-bath to a syrup. The residue is mixed with syrupy phosphoric acid, oling meanwhile, then with 20 to 30 grammes of powdered anhydrous copper lphate and 20 or 25 grammes of fine sand, by which a dry powder is obtained, and is is extracted for about an hour in a Soxhlet with ether dried over anhydrous pper sulphate; the extract is filtered, the copper salt washed with dry ether, the lvent distilled off, the residue taken up in 20 c.c. of water, decolorized with animal arcoal, and its lævo-rotatory power determined. According to Magnus-Levy, the ecific rotatory power of β-hydroxybutyric acid is – 24·12°. Experiments have shown e method to be accurate when tested on urines to which known quantities of the acid had been added; also (after fermentation) on urines containing 5 per cent. of dextrose. Normal urine never contains any substance soluble in dry ether having a lævo-rotatory power.

F. H. L.

The Detection of Peptone in Urine. Z. Cerny. (*Zeit. anal. Chem.*, 592-595.) —The author's experiments confirm the statement of Salkowski, who found that in decolorizing urine with lead acetate a certain proportion of any peptone present was precipitated, and when only present in traces could not be detected in the filtrate by the biuret reaction.

He has found a modification of Hofmeister's method the most satisfactory. On treating the urine with tannin and decomposing the precipitate with barium hydroxide, the filtrates are dark and unsuitable for the biuret test. If, however, the liquid be warmed and shaken so as to introduce air, the colouring matters, including urobilin, are oxidized, leaving a colourless solution.

The precipitates obtained from urine by means of phosphotungstic acid, when treated with barium hydroxide, also yield filtrates which, when oxidized in this way, contain only traces of urobilin. Even when that substance was originally present in considerable quantity, the author had no difficulty in applying the biuret test for peptone.

C. A. M.

Estimation of the Covering Power of Pigments. E. Valenta. (*Oesterr. Chem. Zeit.*, 1901, iv., 533.)—This is a process for estimating the covering power of a pigment which does not require the use of a standard, and which gives results that can be expressed in figures. It depends on the following principle, the pigment being ground into oil or varnish in due proportion: If the colour is applied as a uniform film not exceeding a certain thickness to a sheet of black paper which absorbs all incident light, and if under similar conditions it is applied to a sheet of white paper, the amount of light reflected from a unit surface of the printed black paper is less than that reflected from the same area of the printed white paper. The better the covering power of the pigment, the greater will be the quantity of coloured light reflected from the black printed paper in comparison with that reflected from the printed white paper, since the black paper itself reflects no light, and the white paper all; it only remains, therefore, to measure the reflected coloured light. The pigment is ground with the proper proportion of oil or varnish so as to make a printers' ink, and this is applied by the usual lithographic or "letterpress" system of manipulation to the two pieces of paper. In a suitable colorimeter, such as the author has previously described, a piece of pure (unprinted) white paper is put on one side, and a piece of the printed white paper on the other. The liquid-tube before the former is then charged with so much of a solution of the same colour as the pigment that (neglecting mere brightness) both fields of vision appear equally coloured. As a mean of ten observations the depth of solution is found to be W millimetres. The printed white paper is then replaced by the printed black paper; the light which enters the opposite tube is weakened by altering the reflector, until both fields are equally luminous, and the coloured liquid is adjusted as before to give equal depth of colour, the quantity of solution being S. Assuming hat the inks tested all contain the same proportion of pigment and varnish of known iscosity, and that they are applied in films of equal thickness, the ratio S : W xpressed as a percentage of W gives a figure for representing the covering power of he pigment in absolute terms. For instance, commercial vermilion gave W = 4 millimetres, S = 21 millimetres, whence the covering power was 38·9 per cent. hrome yellow gave 56 millimetres and 35·2 millimetres, or 62·8 per cent. Madder ke ("Krapp") gave 58·6 millimetres and 1·6 millimetres, or 2·7 per cent. Thus he covering powers of chrome yellow, vermilion, and madder lake stand to one nother as 62·8 : 38·9 : 2·7.

Valenta finds that the danger of producing films of varying thickness when sing the printer's roller, rolling-slab, and block (or lithographic stone) is too small be of practical significance, but for extremely accurate work the amount of igment laid on to a unit surface of the papers might be ascertained by the balance, nd due allowance made accordingly. F. H. L.

[It may be observed that Valenta has scarcely laid sufficient stress upon the portance of preparing such inks as he proposes to compare by means of a proper wer-driven mill with chilled iron or granite rollers, or a small laboratory model ereof. The old hand muller and stone is a very treacherous instrument except in ghly-experienced hands; it is impossible to introduce into the oil or varnish a proper oportion of pigment, and, as the microscope shows, it is impossible to reduce that gment to a normally fine state of subdivision. Valenta's last remark is perfectly st; a little experience with the letterpress or lithographic roller and the stone or ock will show that the amount of ink (hence pigment) applied is regulated tomatically, provided the "strength" of the ink is such as to suit the character of e printing press employed. The abstractor has obtained perfectly useful and mparable results, which, however, could not be expressed in figures by merely ting the proportion of vehicle needed to make an ink of proper strength from a ven pigment and a standard varnish on a certain mill, and then by examining the aracter of the impression produced in a hand-press, using a standard "half tone" ock very fine in the grain, and having spots of solid colour as well as patches of ure paper. Impressions made in this way can be filed in the dark and kept for ference; they seem to last unchanged for a long time, and they undoubtedly form n efficient means of judging the relative value of any new pigment sample as regards vering power and effect (if any) on the drying power of linseed oil.] F. H. L.

INORGANIC ANALYSIS.

The Determination of Traces of Antimony in the Presence of Large uantities of Arsenic. G. Denigès. (*Bull. Soc. Chim.*, 1901, xxv., 995-1000.)— *Deposition by means of Tin-platinum Couple.*—The substance under examination .g., mirror obtained in Marsh's test) is dissolved in a very small quantity of nitric cid in a round-bottomed platinum crucible, the solution evaporated to dryness on he water-bath, and the residue dissolved in dilute hydrochloric acid (1 : 4), so as to ive a solution containing not more than 5 milligrammes per c.c.

A strip of tinfoil, about 4 to 5 centimetres long and 1 centimetre broad, with a pointed end, is then placed in the crucible, with the point resting on the centre of the bottom of the crucible, and its other end resting on the edge.

It is advantageous to concentrate the liquid to a tenth or even a twentieth of a c.c. After ten to thirty minutes, according to the quantity of antimony, the crucible is washed with water, alcohol, and ether, allowed to dry spontaneously, and examined for a stain.

Arsenic acid under the same conditions does not produce any stain when the quantity of arsenic is not more than 5 milligrammes per c.c., provided the time of contact does not exceed thirty minutes. Above that proportion, arsenic gives a stain, though much more slowly than antimony. The author finds that it is thus possible to detect 0·002 milligramme of antimony in the presence of 125 times that quantity of arsenic.

For a quantitative estimation, the stain is compared with that obtained under the same conditions with solutions of antimony in hydrochloric acid containing 2, 4, 8, 12, 16, and 20 thousandths of a milligramme in 0·1 c.c.

Zinc is much less sensitive than tin for this test, and cannot be used in the presence of arsenic.

2. *Formation of Cæsium Antimony Iodide.*—One gramme of potassium iodide and 3 grammes of cæsium chloride are dissolved in 10 c.c. of water, and 1 drop of dilute ammonia (1 : 10) added to the solution. On adding this reagent to a solution of antimony in hydrochloric acid (1 : 4) or sulphuric acid (1 : 10), containing at least 2 milligrammes per c.c., a red precipitate of the double iodide is instantaneously produced, the form of which, in the absence of bismuth, is frequently characteristic of antimony.

It is possible to detect the hexagonal crystals under the microscope in a sulphuric acid solution of antimony not containing more than 0·0001 milligramme in $\frac{1}{100}$ c.c. The reaction is less sensitive in a hydrochloric acid solution.

This quantity of antimony can also be detected in the presence of 500 times the quantity of arsenic, provided that the amount of the latter does not exceed 5 milligrammes in 0·1 c.c. Above that proportion free iodine begins to be liberated.

In examining the stains on the platinum crucible in the first test, or Marsh's mirrors, a drop of nitric acid is added to the substance in a porcelain crucible, and evaporated to dryness. A drop of the cæsium reagent is then placed upon the dry residue, together with an equal volume (0·01 c.c.) of dilute sulphuric acid (1 : 10). After a short time, when antimony is present, a reddish layer is formed, which does not disappear on adding a small drop of sulphurous acid solution, and the intensity of which is proportional to the amount of antimony present.

If the quantity of antimony in the residue is as much as 0·01 milligramme, it may be dissolved in 0·1 c.c. of hydrochloric acid (1 : 4) and a small drop of the solution, mixed with the same quantity of the cæsium reagent on a glass slide, which is examined after two minutes under the microscope. C. A. M.

A Method for the Quantitative Separation of Nickel and Zinc. Arthur osenheim and Ernst Huldschinsky. (*Ber. Deutsch. Chem. Ges.*, xxxiv., 16, 13.)—A few months ago the authors published a method for the separation of balt and nickel based on the solubility in a mixture of ether and amyl alcohol of e compound ammonium cobalt thiocyanate (*Ber.*, xxxiv., 2050). They have now nd that a similar method cannot be used to separate zinc from cobalt, since zinc o forms an analogous compound, soluble in the various organic solvents, but that nsequently zinc can be separated from nickel in the same way as cobalt. For this rpose, the solution of the chlorides of the two metals is evaporated to dryness, e residue dissolved, together with 12 grammes ammonium thiocyanate, in not ore than 50 c.c. water, and the solution repeatedly extracted in a Rothe apparatus th a mixture of 1 volume amyl alcohol and 25 volumes ether. From the lution containing the zinc the ether and alcohol are distilled off, the residue is ssolved in hydrochloric acid, and zinc precipitated with sodium carbonate. The ueous nickel solution is evaporated to dryness, the residue heated to redness, and ckel estimated electrolytically after dissolving in nitric acid and converting into e double ammonium oxalate.

Analyses by this method of solutions containing known quantities of zinc and ckel gave good results. Some alloys containing copper, nickel, and zinc were also alysed by this method, as well as by separating nickel and zinc with hydrogen lphide in acetic acid solution, and the results were found to agree well with each her (within 0·2 per cent.), the new method of separation being far more expedious than the old.

In using the method, it is to be noted that nitric acid must be completely moved before the extraction, as it renders the latter very difficult to carry out. mall quantities of free hydrochloric acid left after evaporation may be neutralized ith dilute sodium hydroxide solution. Any iron or cobalt in the alloy will be found ith the zinc, and may be recognised by the red or blue colour of the extract. This nstitutes a disadvantage of the method, since for technical purposes it is very often ly necessary to find the total amount of nickel and cobalt. However, the small antities of cobalt occurring in these alloys can be readily and accurately separated the potassium nitrite method after weighing the zinc oxide. A. G. L.

A Reagent for Ferrous Salts and for Certain Metals of the Zinc and Iron roup. G. Denigès. (*Bull. Soc. Pharm. Bordeaux*, 1901, xli., 161; through *Journ. harm. Chim.*, 1901, xiv., 530, 531.)—The author's test is based upon the fact that loxan, or derivatives containing the alloxan group, give a blue coloration with kaline solutions of ferrous salts.

The reagent is prepared by mixing 2 grammes of uric acid and 2 c.c. of nitric id (40° Bé.), adding 2 c.c. of water when the reaction begins to slacken, heating ntly until a clear solution is obtained, and diluting it to 100 c.c.

A few c.c. of this reagent mixed with a trace of a solution of a ferrous salt, and ndered alkaline with potassium or sodium hydroxide, gives a blue coloration, which changes to light yellow on shaking the tube, owing to the conversion of the ferrous into a ferric salt.

The reagent can also be used to identify certain metals in the metallic state. Thus, a fragment of zinc boiled for some time with 2 to 3 c.c. of the liquid gives a yellow coloration. Under the same conditions magnesium gives a deep carmine colour; cadmium a grenadine tint; iron a yellowish-brown tint; nickel (rapidly) and cobalt (more slowly) an orange shade; and manganese (slowly) a deep carmine red colour.

Any confusion between these different colours is prevented by the addition of 1 or 2 drops of sodium hydroxide solution, when the following colorations are produced: Carmine with zinc and cadmium; violet with magnesium; bluish-violet with manganese; blue with iron; Bordeaux red with cobalt; and sepia and then red with nickel.

The author attributes these reactions to the formation of metallic purpurates in the following manner: The hydrogen liberated by the action of the nitric acid on the metals reduces the alloxan to alloxantin, and also converts part of the nitric acid into ammonium nitrate, thus producing the necessary substance for the formation of purpuric acid.

C. A. M.

New Method for the Estimation of Manganese. G. v. Knorre. (*Zeits. f. angew. Chem.*, xiv., 1149.)—In Hampe's method (ANALYST, 1885, 191) there is danger of loss in consequence of the very energetic reaction between 1·4 nitric acid and potassium chlorate. The author therefore oxidizes with ammonium persulphate. (See also ANALYST, xxvi., 221.)

A solution of the ore or alloy is made, containing a little free sulphuric acid and no nitric or hydrochloric acid. An excess of persulphate is added, and the liquid is boiled for four to seven minutes. The precipitated manganese dioxide is filtered off and washed. The filter is placed in a flask, and the precipitate is dissolved by adding an excess of standard ferrous sulphate solution, which is then titrated back with standard permanganate. It is best to standardize the permanganate on a pure manganese compound under the same conditions as occur in the actual determination.

A. M.

Volumetric Estimation Manganese. H. Ramage. (*Chem. News*, lxxxiv., 209.)—For wrought iron, steel, and pig-iron, weigh out 1·1 gramme, add 30 c.c. of dilute nitric acid (about 5N), and boil. Decant the solution, filter off carbonaceous matter if necessary, and to the part undissolved add more nitric acid up to 25 c.c. The free acid is then about 4N. If the sample contains much silicon, care must be taken when boiling not to unduly concentrate the acid, or the silicic acid will separate in a form which blocks up the filter. Cool to about 16° C., oxidize with 2 grammes of "sodium bismuthate," stir for three minutes, and filter through an asbestos filter. Add hydrogen peroxide solution (approximately $\frac{N}{10}$ in N nitric acid) from a burette until the *reddish* colour disappears, then add 1·5 to 3·0 c.c. in excess, and titrate with $\frac{N}{10}$ permanganate. The hydrogen peroxide solution is standardized

nst the permanganate in the presence of $\frac{N}{4}$ nitric acid. Each c.c. of $\frac{N}{10}$ hydrogen
xide solution reduced equals 0·1 per cent. of manganese. A determination can be
pleted in twelve minutes.

In the case of ferromanganese and spiegels the process given by Reddrop and
uthor (*Journ. Chem. Soc.*, 1895, 268) need not be modified.

A large excess of hydrogen peroxide must not be used, as a reaction takes place
een it and the iron. For each c.c. excess, 0·1 c.c. is reduced immediately.
her small error in the opposite direction is due to reduction of the permanganic
during and after filtration. This may be avoided by more complete oxidation of
original solution in nitric acid; to effect this it is boiled with 0·5 gramme of
uthate, cooled, and excess of sulphurous acid is added, then 1·5 gramme of
uthate, and the determination is completed as described above. A. M.

Volumetric Estimation of Manganese. F. Ibbotson and H. Brearley
m. News, lxxxiv., 247.)—In the "bismuthate" process (see above Abstract) the
ction of the permanganic acid during and after filtration is ascribed by the
ors to the presence of carbon. This makes it essential to complete the oxidation
e carbon in hot solutions—for high carbon steels at any rate. Other substances
ently present in steel-making alloys interfere with the process; some of these
pecially considered. The process generally used by the authors is as follows:
olve 1·1 gramme of the metal in 35 c.c. of 1·20 nitric acid, and then add
uthate a little at a time to the somewhat cooled solution until a permanganate
r persists, or on boiling is decomposed to manganic oxide. Clear up the
anganate or the dioxide precipitate with a little hydrogen peroxide, sulphurous
or ferrous sulphate free from manganese. Cool the solution, and add about
c. of water and a considerable excess of bismuthate. Filter, wash with dilute
4 per cent.) nitric acid, and titrate with decinormal permanganate.

Unlike hydrogen peroxide, the excess of ferrous sulphate does not react with
nitrate, and is therefore preferable. It can be safely used in cold nitric acid
ions as above, if the titration is not needlessly delayed. Molybdenum, titanium,
vanadium do not interfere in this modification as they do in Ramage's process.
romium be present, it is liable to be slowly but completely oxidized by
uthate to chromic acid, thus giving high results for manganese. The bismuthate
d therefore be added, and the solution shaken and filtered quickly. The
nce of tungsten introduces no error; but in steels containing much tungsten,
particularly in chrome tungsten steels, the precipitate has a tendency to pass the
The joint presence of tungsten and hydrofluoric acid causes the results to be
and very erratic, so the hydrofluoric acid should be driven off with sulphuric
A. M.

Volumetric Estimation of Manganese. L. Dufty. (*Chem. News*, lxxxiv.,
—A simplification of Reddrop and Ramage's process (see above) for use in
s. The nitric acid solution, which has been used for determining the carbon
rtz), is transferred to a 25 c.c. stoppered test mixer and made up to a definite

volume. A standard steel of known manganese content is treated in like manner. Equal quantities of bismuthate (0·2 gramme) are added, the contents mixed, allowed to settle (thirty minutes), measured quantities of the clear pink solutions are transferred to stoppered carbon tubes, and the colours compared in the usual manner. (See also ANALYST, this vol., p. 27.) A. M.

A New Gravimetric Method of Estimating Tellurium. A. Gutbier. (*Berichte*, 1901, xxxiv., 2724-2726.)—The ordinary methods in which the tellurium is precipitated from an acid solution is open to the objection that the element readily undergoes oxidation, and may then dissolve to a considerable extent during the washing. On the other hand, when the precipitation is effected by boiling an alkaline solution with dextrose, the excess of alkali necessary interferes with the determination.

The author's method consists in precipitating the tellurium with hydrazine hydroxide, by which method oxidation of the tellurium does not occur.

In making a determination, a weighed quantity of telluric acid was dissolved in warm water in a dish covered with a glass, and a 10 to 20 per cent. solution of hydrazine hydroxide introduced by means of a pipette through the opening between the spout and cover of the dish, until the liquid became dark blue or black, and on boiling yielded a flocculent precipitate of metallic tellurium. A further quantity of the hydrazine hydroxide was now added in successive small portions until the supernatant liquid no longer became coloured. After each addition the dish was heated until the tellurium had completely settled, leaving a colourless liquid above.

When cold the liquid was filtered through a paper filter previously dried at 105° C. and weighed, and the precipitate washed with hot water and dried on the filter at 105° C. until constant in weight. Care was taken to do the washing and drying as rapidly as possible, in order to avoid any chance of oxidation.

In test experiments a sample of telluric acid containing theoretically 55·50 per cent. of tellurium gave by this method 55·29 to 55·70 per cent. of tellurium.

C. A. M.

The Determination of Cæsium and Rubidium as Bisulphate, and of Potassium and Sodium as Pyrosulphate. Philip F. Browning. (*Zeits. Anorg. Chem.*, xxix., 141.)—The author finds that cæsium and rubidium salts of volatile acids, when treated with an excess of sulphuric acid and heated at a temperature of 250° to 270° to constant weight, yield the acid salts $CsHSO_4$ and $RbHSO_4$. Cæsium shows a slight tendency to retain a little more sulphuric acid than corresponds with the acid salt, but if this is heated above 300° it loses water as well as sulphuric acid. When sodium and potassium salts are treated in the same way they yield the pyrosulphates $Na_2S_2O_7$ and $K_2S_2O_7$. Lithium salts yield neither the bisulphate nor the pyrosulphate. The results quoted in support of the above statements are fairly satisfactory. A. G. L.

Methods for Examining the Recovered Soda of Cellulose Factories. G. Lunge and W. Lohöfer. (*Zeits. f. angew. Chem.*, xiv., 1125.)—This product differs from the crude soda obtained by the Leblanc process in that it contains large quantities of sulphide and also, if straw be used as the source of cellulose, considerable quantities of silicate. For regulating the factory processes it is necessary that the following constituents should be estimated: Caustic soda, carbonate, silicate, sulphide, sulphite and sulphate of sodium, and insoluble matter. It is not possible to entirely precipitate the silicate along with the carbonate by the addition of barium chloride; consequently, other methods must be used to distinguish the constituents one from the other. The authors find that in the presence of sodium chloride sodium silicate requires the full theoretical quantity of hydrochloric acid to neutralize it, whether methyl orange or phenolphthalein be used as indicator. The analysis is carried out as follows:

Fifty grammes are dissolved at about 45° C. by shaking with about 500 c.c. of water, free from carbonic acid and air, in a closed litre flask. This is then filled to the mark.

1. *Insoluble* matter is determined as is usually done in crude soda.

2. *Alkalinity.*—Twenty c.c. is titrated with $\frac{N}{5}$ hydrochloric acid, using phenolphthalein as indicator; then methyl orange is added, and the titration is continued. The end-point is sharp.

3. $Na_2S + Na_2SO_3$.—Twenty c.c. are diluted with air-free water to about 200 c.c., acidified with acetic acid, and rapidly titrated with $\frac{N}{10}$ iodine solution, using starch as indicator.

4. Na_2SO_3.—Alkaline zinc solution is added to 100 c.c. of the solution to precipitate the sulphide. The liquid is poured through a dry filter, 50 c.c. of the filtrate are acidified with acetic acid, and titrated with $\frac{N}{10}$ iodine.

5. Na_2SiO_3.—Twenty c.c. are evaporated to dryness with hydrochloric acid, and silica is determined in the usual way.

6. Na_2SO_4 is determined by precipitation with barium chloride.

The alkalinity determined with methyl orange gives

$$Na_2CO_3 + NaOH + Na_2SiO_3 + Na_2S + \tfrac{1}{2}Na_2SO_3,$$

whereas the number obtained with phenolphthalein indicates

$$\tfrac{1}{2}Na_2CO_3 + NaOH + Na_2SiO_3 + \tfrac{1}{2}Na_2S.$$

Twice the difference therefore gives $Na_2CO_3 + Na_2S + Na_2SO_3$. Subtracting (3) from this gives Na_2CO_3. The difference between (3) and (4) gives Na_2S. All the compounds which go to make up the alkalinity are now known excepting NaOH, which can therefore be calculated by difference. A. M.

The Analysis of Alkali Persulphates. G. Allard. (*Journ. Pharm. Chim.*, 1901, xiv., 506-508.)—A common method of valuing alkali persulphates is based upon the fact that they liberate iodine from potassium iodide and on the titration of this iodine with standard thiosulphate solution.

From the author's experiments, which are given in detail, it appears that if sulphuric acid be added to the liquid, as advocated by Rapp and by Moreau, the results are considerably too high, whereas in a neutral solution they are accurate. The reaction is complete after thirty minutes, but it is advisable to wait for an hour before titrating. C. A. M.

Quantitative Determination of Fluorine in Fluorides Easily Decomposable by Sulphuric Acid. W. E. Burk. (*Journ. Amer. Chem. Soc.*, xxiii., 825.)—The following is a modification of Carnot's method (*Compt. rend.*, 1892, 114, 750) for the estimation of fluorine. An intimate mixture of 0·2 gramme of the fluoride and 3 grammes recently calcined powdered silica is decomposed at a temperature of 120° to 135° by means of 40 c.c. of sulphuric acid. The evolved gases are led through three U-tubes, plugged at intervals with glass wool, and cooled by means of water, which serve to intercept any sulphuric acid fumes, to the bottom of a narrow cylinder, containing a layer of mercury, and above this 20 c.c. of a 10 per cent. solution of potassium fluoride, which converts the silicon fluoride into potassium fluosilicate.

Immediately before use a current of dry air is drawn through the whole apparatus, which is at the same time warmed. After the introduction of the sample and sulphuric acid, which latter should previously be heated to 165° whilst a current of air is passing through it, the decomposition flask is heated for one and a half hours, the current of air being maintained all the time. The solution and precipitate in the receiving vessel are then transferred to a beaker by means of a pipette, and the vessel rinsed out with small quantities of water. The total volume should not exceed 75 c.c., and a thin coating on the inner wall of the receiving vessel should not be removed, as it is due to addition products of the potassium fluoride and the glass. After adding an equal volume of alcohol, the precipitate is allowed to subside for half an hour, filtered, washed with alcohol, dried at 100°, and weighed. No test analyses are given. A. G. L.

A Gas Volumetric Method of Determining Chlorides, Hydrochloric Acid, Silver, and Phosphates. E. Riegler. (*Zeit. anal. Chem.*, 1901, xl., 633-638.)—*Chlorides.*—The principle of the method is that on treating silver chloride with hydrazine sulphate and sodium hydroxide, the silver separates in the metallic form whilst nitrogen is liberated; thus

$$4AgCl + N_2H_4H_2SO_4 + 6NaOH = 4Ag + 4NaCl + Na_2SO_4 + 6H_2O + N_2.$$

According to this equation, 1 part of nitrogen corresponds to 20·424 parts of silver chloride, but in practice the author found that the corresponding quantity was only 20·2 parts.

In making a determination, the solution of the chloride, which should not yield more than 1 gramme of silver chloride, is acidified with 2 to 3 c.c. of nitric acid, and treated with a 5 per cent. solution of silver nitrate. The resulting precipitate is collected, washed, and transferred to the outer vessel of a Knop-Wagner nitrometer, into which are also introduced about 30 c.c. of water, and about 0·5 gramme of crystallized hydrazine sulphate, whilst 10 c.c. of a 10 per cent. solution of sodium hydroxide are placed in the inner vessel.

The nitrogen is then liberated, measured, with the usual precautions as to temperature, etc., and calculated into its weight in milligrammes. This weight, multiplied by 20·2, gives the corresponding weight of silver chloride, whilst the factor 5 gives the amount of chlorine, and the factor 8·23 the amount of sodium chloride.

A phosphate can be converted into the silver salt, and this into the chloride, which is determined in the above way. C. A. M.

The Detection of Chromic Acid by means of Hydrogen Peroxide in the Presence of Vanadic Acid. C. Reichard. (*Zeit. anal. Chem*, 1901, xl., 577-586.) —One of the sharpest tests for chromic acid and its salts is the well known blue coloration of perchromic acid (Cr_2O_7) which is obtained with hydrogen peroxide.

The author has made a series of experiments which show that the presence of vanadic acid has a strong inhibitive influence on the production of this reaction.

Even when potassium bichromate is added in the proportion of 10 parts to 1 of ammonium vanadate, the restrictive influence of the vanadic acid is still perceptible. By adding sodium arsenate or sodium phosphate, however, the effect of vanadic acid on the perchromic acid reaction is overcome.

As regards other salts, it appears that sodium nitrate, or nitrite, ammonium persulphate, potassium iodate, or uranium nitrate, have no perceptible restrictive influence on the vanadic acid.

The presence of certain acids allied to vanadic acid (molybdic and tungstic acid) were found to exert a similar restrictive influence on the perchromic acid reaction. C. A. M.

REVIEWS.

FERMENTS AND THEIR ACTIONS. By CARL OPPENHEIMER, M.D., Ph.D. Translated from the German by C. AINSWORTH MITCHELL, B.A., F.I.C. London: Chas. Griffin and Co. Price 7s. 6d. net.

This work contains, within the compass of some 300 pages of reading matter, a résumé of almost all that has been so far published on the subject of enzymes. The matter is therefore presented to the reader in very condensed form, but without any apparent sacrifice of lucidity. The amount of literature which has been perused by the author in the preparation of the book is simply enormous; there is scarcely a page without a reference to some work or periodical, and to some pages as many as a dozen are appended. The book is divided into a general and special part; the former contains a brief historical notice of the enzymes, the definition of these bodies, the influence of external agents upon them, the mode of action of the enzymes, their secretion in plants and animals, and their importance to life. The special part deals with the enzymes individually, dividing them into two great classes—hydrolytic and oxidizing. In this latter class Buchner's zymase is included, the author being of opinion that the oxidation in this case is intromolecular. All through the book Dr. Oppenheimer treats the denominations "ferment" and "enzyme" as synonymous, and as this clashes somewhat with the commonly-accepted notions of the two terms, it may prove somewhat confusing if not borne in mind. An excellent innovation is the author's proposal to derive the name of the enzyme from that of the body on which it acts; thus the enzyme that converts maltose into glucose, and which has hitherto been called glucase, is now termed maltase, ordinary diastase amylase, etc. The volume concludes with a copious bibliographical index of the literature of the enzymes; it gives references to no less than 1,270 books or papers, and is followed by a general index of subjects, and also one of authors' names. Dr. Oppenheimer deserves our warmest thanks for bringing such an immense mass of information into such readily accessible form; the book cannot fail to be of extreme value to the student as well as to the expert. His views on some of the problems connected with enzymic action will be read with deep interest, although it is painfully evident that finality is a long way from being reached in this recondite subject. The translator, Mr. C. A. Mitchell, is to be congratulated on the excellent manner in which he has performed his by no means light task of translating; he has also interpolated a number of additions to the original text in order to bring the work up to present date. In his preface he calls attention to the recent discovery that some of the enzymes possess a reversible action, and this somewhat contravenes some of the views of the author on the theory of enzymic action. W. J. S.

American Handy Book of the Brewing, Malting, and Auxiliary Trades. By Robert Wahl, Ph.D., and Max Henius, Ph.D. Chicago: Wahl and Henius. Price $10.

This work, as stated in its preface, is designed for a book of ready reference, and makes no pretence to be a text-book on brewing. It consists of 1,266 pages of matter, clearly printed on very thin paper, bound in limp leather, so that it may be carried in the pocket. The authors have been assisted in the production of the book by a number of assistant editors and contributors. The scope of the work is wide indeed, for it not only contains chapters on such subjects as chemistry, brewing materials, micro-organisms, etc., which are ordinarily met with in books on brewing, but such cognate subjects as mensuration, mechanics, machinery, physics, legal information, etc. There is a copious bibliography of works on brewing and kindred subjects, a very useful English-German dictionary of technical terms, and a very full and complete general index. The book is a veritable *multum in parvo*, and certainly well fulfils the object for which it is intended. It is the first standard book that has appeared on American brewing, which, though originally derived from the German system of lager-beer brewing, has developed an individuality peculiar to itself; and the same may be said of American top-fermentation brewing. Though primarily intended for the American brewer, it contains much that is of interest and utility to brewers of other lands. W. J. S.

Sewage and the Bacterial Purification of Sewage. By Samuel Rideal, D.Sc. (Lond.). The Sanitary Publishing Co., Ltd. Price 14s.

The appearance of a second edition of this work within a few months of the first affords a convincing proof that it meets a widespread want. Nor is the reason of its popularity far to seek. When it is considered that treatises on this subject have in general been the work of enthusiasts eager to inform the world of the disivery of the universal remedy for pollution; or of engineers, able and resourceful, it true to their creed of removal rather than depuration; or, lastly, of plodding iekers after information who, with the best intentions, have made confusion worse onfounded by heaping in disorder the ill-digested accumulations of tours of inspec- on, it is not astonishing that a well-considered account, such as that under present otice, of sewage, the changes it undergoes in its natural conversion into inoffensive water," and the artificial means that have been devised to facilitate and cheapen iat conversion, should obtain a world-wide acceptance. The present time is, of ourse, exceptionally opportune for the appearance of a work of this description. he enormous advances of the past few years, not only in the knowledge of the tionale of sewage purification, but in the technical application of that knowledge, gether with the official fixation of established progress by the periodical issue of the ports which may now be expected from the Royal Commission, all tend to render elcome the publication of a scientific statement of the latest views of both theory nd practice.

After a preliminary quotation of the findings of the Royal Commission as sum- iarized in their interim report, the author proceeds in the earlier chapters to define ewage, and to describe methods for its chemical and bacterioscopic examination, ie preference being given to methods which, if not of the highest degree of accuracy, re capable of easy and rapid application, a matter of prime importance in this articular connection. The various standards of purity of effluents also receive ue notice.

Chapter V. introduces the subject proper, the nature of the chemical changes iduced in sewage by bacteria, and is followed by a section on Irrigation and Sewage arms. Chemical precipitation, forming the chief subject of Chapter VII., would eem somewhat out of place in a work devoted to bacterial purification, but follows aturally upon a brief reference to simple subsidence, which is now very generally ound to be an economical preliminary to the true bacterial treatment. Chapter VIII., n Sterilization, gives some idea of the task before the Royal Commission in the ursuit of a method of "eliminating or destroying organisms, or at least those giving ise to infectious diseases."

The concluding chapters describe in detail the various appliances that have been evised for assisting the bacteria in their beneficent labours, by providing the con- itions under which each group of species can work to the best advantage: and erhaps the most useful feature in the whole book is the clear and emphatic manner n which the absolute necessity for the appropriate alteration of conditions in con- ormity with the stage of purification at which the sewage has arrived is insisted on. his has long been a matter of course in such industries as depend on the successive tilization of bacteria of different types and habits, and there can be no question that he progress of sewage treatment has been seriously retarded by the failure, except n certain isolated instances, to realize this truth.

A brief account of trade wastes and their influence on bacterial action brings to close a work which will assuredly take, if it has not already taken, a position as he standard authority on the subject, and which no one responsible for the con- truction of sewage disposal works can afford to leave unread. F. W. S.

OBITUARY NOTICE.

THE LATE DR. G. HARRIS MORRIS.

It is with sincere regret that we have to record the death of one of our members, Dr. G. Harris Morris, who passed away on December 30 of last year, at the early age of forty-three years. Born at Bristol, he commenced his scientific career as assistant to Dr. Tilden at Clifton College. He then proceeded to Germany, studied chemistry under Dr. Wislicenus, and graduated as Ph.D. at Würtzburg. On his return to England he rejoined Dr. Tilden at Clifton, and when the latter was appointed Professor of Chemistry at Birmingham, accompanied him there, and acted as his demonstrator. In 1883 Dr. Morris was appointed chemist to Messrs. Worthington of Burton, and in conjunction with Mr. (now Dr.) Horace Brown continued those valuable researches on the "Action of Diastase on Starch," which had been commenced some years previously by Brown and Heron. These were followed in 1890 by a masterly investigation (also in conjunction with Brown) on the "Germination of the Grasses," and subsequently by a similar investigation into the physiological chemistry of foliage leaves. More recently, in conjunction with Brown and Millar, a paper of great value and interest was communicated to the Chemical Society—"On the Experimental Methods of the Investigation of Starch Conversion Products," which placed the methods of analysis of these bodies on a more certain foundation. Besides these, Dr. Morris contributed a number of papers to the Chemical Society and to the Institute of Brewing. He was conjoint author with Dr. Moritz of that well-known work "A Text-book of the Science of Brewing." Dr. Morris has for some years past been engaged in private practice as partner with Dr. Moritz. He also held the positions of editor to the *Brewing Trade Review*, and analyst and scientific adviser to the Country Brewers' Society. For some time he was lecturer on bacteriology at the Jenner Institute. Last year the University of Birmingham conferred on Dr. Morris the honorary degree of B.Sc.

INSTITUTE OF CHEMISTRY OF GREAT BRITAIN AND IRELAND.

The following is a list of the names of the candidates who passed the Intermediate Examination of the Institute of Chemistry held in January, 1902: G. J. Alderton; K. B. Benham; J. L. Garle; H. W. Hill; J. Johnston; A. W. Knapp, B.Sc. (Birm.); S. G. Paine; W. B. Parker; J. B. Pursglove; J. C. Sheldon; H. A. Tempany; and R. Tyler.

Final A.I.C. Examination.—Branch "A" (Mineral Chemistry): A. G. Armstrong; W. W. Lumsden; A. B. Shepherd, B.Sc. (Vict.); and W. F. Sutherst, Ph.D. (Geneva).

Branch "D" (Organic Chemistry): G. Clarke, jun.; E. D. M. Neumann, B.A. (Oxon.), Ph.D. (Göttingen); H. A. D. Neville, B.Sc. (Lond.); G. M. Norman, A.R.C.Sc., B.Sc. (Lond.); W. H. Nuttall; W. H. Peters; and V. W. Theobalds.

Branch "E" (Analysis of Food and Drugs, including an Examination in Therapeutics, Pharmacology, and Microscopy): F. W. F. Arnaud; J. Evans; J. E. Jenkins; W. Partridge; S. O. Richmond; and E. C. Spurge.

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VOLUME XXVII.—No. 312.
MARCH, 1902.

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THE ANALYST.

MARCH, 1902.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

ANNUAL DINNER OF THE SOCIETY OF PUBLIC ANALYSTS.

THE annual dinner of the Society took place on Wednesday evening, February 12, at the Hotel Cecil. The President, Dr. J. Augustus Voelcker, M.A., B.Sc., presided. About sixty members and guests were present, among the latter being Sir Ernest Clarke, M.A., Secretary of the Royal Agricultural Society of England; Mr. T. H. Elliot, C.B., Secretary of the Board of Agriculture; Mr. John Lithiby, Assistant Secretary of the Local Government Board; Dr. T. E. Thorpe, C.B., F.R.S., Principal of the Government Laboratory; Mr. W. R. Bousfield, K.C., M.P.; Dr. Henry E. Armstrong, F.R.S.; Dr. J. H. Gladstone, F.R.S.; Professor William A. Tilden, D.Sc., F.R.S.; Professor J. Millar Thomson, LL.D., F.R.S., President of the Institute of Chemistry; Mr. G. T. W. Newsholme, President of the Pharmaceutical Society; Mr. Charles Hawksley, President of the Institution of Civil Engineers; Dr. Rudolph Messel; Dr. Squire Sprigge; Dr. Arthur F. Voelcker; Dr. J. C. Philip; Mr. Thos. E. Holmes, M.B.; and Mr. Aubrey W. Rake. The Right Hon. R. W. Hanbury, M.P., President of the Board of Agriculture, was prevented by indisposition from being present.

The following toasts were proposed: "His Majesty the King," and "Her Majesty Queen Alexandra, their Royal Highnesses the Prince and Princess of Wales and other Members of the Royal Family," by the President; "The Houses of Parliament," by Mr. W. W. Fisher, M.A., responded to by Mr. W. R. Bousfield, K.C., M.P.; "Government Departments," by Mr. Otto Hehner, responded to by Mr. T. H. Elliott, C.B., and by Dr. T. E. Thorpe, C.B., F.R.S.; "The Society of Public Analysts," by Professor William A. Tilden, D.Sc., F.R.S., responded to by the President; and "The Guests," by Mr. Bertram Blount, responded to by Sir Ernest Clarke, M.A., and by Dr. Henry E. Armstrong, F.R.S.

THE PRESIDENT'S ANNUAL ADDRESS.

(*Delivered at the Annual Meeting, January* 22, 1902.)

In following, as I do, in the presidential chair those, my predecessors, who were, one and all, closely connected with the work and the relations of the Public Analyst in the official and limited sense of the term, I cannot fail to have noticed, associated with my selection for the office, features both of advantage and disadvantage. In the first place, I can regard my appointment to this position of honour as a mark of the growing expansion of this Society, of the widening of its bounds, the enlargement of its interests, and the nearer realization of the first of its objects as defined in its constitution. The first object of the Society is therein stated to be "to cultivate the study of analytical chemistry, by the holding of periodical meetings, and by the publication of a journal devoted mainly to analytical chemistry." It was well, indeed, and, in fact, almost necessary, that the class who should first band themselves together, for their common interests, should be those whose official positions brought them into relation with one another and the public, and hence it is that the earlier days of the Society were more essentially those of the Public Analyst in the restricted sense, and that the papers read and the discussions held were mainly those concerned with the official work of the then newly-created and, I am bound to say, somewhat imperfect and frequently-abused individual—the Public Analyst. But, as time has gone on, so have individuals improved and gained by experience, so have methods improved and new ones been devised, and the Public Analyst of to-day is no longer the Public Analyst of twenty, or even ten, years ago. More than this, it has been realized, both within this Society and to an increasing extent outside it, that a man to be a Public Analyst in the true sense must be not only something more than a mere machine, capable of evolving certain analytical results by stereotyped methods, but that he requires to be in every sense a chemist by training, and to have this enforced by subsequent practical experience; that he is a thinking and reasonable being; and that he is an impartial and valuable servant appointed in the public interest. With this change has come about also a change in the attitude of the Public Analyst himself. He finds it necessary that he should be not merely the master of a certain routine of work of limited application, and be able to analyse and talk about the analysis of a limited number of common articles like milk, butter, and spirits, but he finds, too, that he has to become an analyst in the wider sense, and that his real object must be the same as that of the Society—"to cultivate the study of analytical chemistry." Hence it is that anyone now turning over the pages of the official journal of the Society, the Analyst, or looking at the names of the members of the Society, will find a wider sphere now embraced, and that, with all due deference to and appreciation of the valuable work done by our old friend Dr. Vieth, and since ably carried on by his successor, Mr. Richmond, the discussions of the Society have travelled far beyond the perennial topic of "milk." From being a Society, as originally formed, to further the interests and knowledge of a special class, the Society of Public Analysts has felt that its members can no longer have limited views, but must be analytical chemists in the wider sense, and have interests in common with all those who devote themselves to this branch of chemical science. This has been well reflected in the ANALYST, which is the one journal in this country that is devoted to the advancement of analytical chemistry, and which holds now an accepted and acceptable position among the scientific literature of the day. The papers read before the Society, as the list that follows will tell, and the discussions which have taken place on them, show the same broadening of interest, and I may lastly look upon your selection of myself as your President as an evidence of this. Not myself holding any appointment as Public Analyst under the Sale of Food and Drugs Act, I may yet claim to be a Public Analyst in the wider sense to which you, in your development of the aims and interests of the Society, have given expression, and it was this knowledge that led me cheerfully to accept the honour you wished to put upon me.

But while there may have been, thus far, an advantage to the Society, I feel that there has been a corresponding disadvantage in the departure that has been made—now for the first time. My not being a Public Analyst has, I fear, thrown me, in some respects, out of touch with your particular needs, and out of full appreciation of the often difficult duties you are called upon to discharge. But let me say that you, at least, have not let this be apparent to me, and I have now passed through my first term of office with a deeper sense than ever I had of the ability, the value, the necessity, and the conscientiousness of the Public Analyst as a class. Not that there is not still much that might be improved, that a higher standard of professional conduct and of consideration one for another might not be cultivated, that there might not be less of self-seeking and tendency to underbid one another, and to forget what is due to the profession, in the desire to affix this or that appointment. But these are matters which cannot be improved by the enforcing of any strenuous regulations, or by entering into any compact, but which must be left to the growth of a better feeling and of a higher sense of honour among professional men, and also largely to the force of example.

It has been, then, my genuine pleasure to preside this past year over your deliberations, to take my part in furthering your endeavours, and to do what I could towards maintaining the dignity of the office held by my distinguished predecessors, and to improving the position of the analytical chemist in the eyes of the public and in his official relations.

As regards the Society itself, there has been no marked accession of members, for only nine fresh names have been added, as against twenty in 1900, while the removals have been seven in number—viz., three by death, two by resignation, and two on account of non-payment of subscription. It is, on the other hand, matter for satisfaction that the new list of members standing for election at present contains six fresh names. By death the Society has lost three members, Mr. Norman Leonard, Mr. E. W. Martin, and Dr. G. Harris Morris. Mr. Leonard had long been known to us, and was a frequent contributor to the pages of the ANALYST. He held the post of Public Analyst for Northampton, and was closely associated with our respected ex-President, Dr. Thomas Stevenson. Mr. Martin was one of our American members, and so less known to us personally; but in Dr. Morris we lose one whose work was well known, and for whom, especially in more recent days, we felt a special sympathy. In the domain of plant physiology Dr. Morris was for many years associated with Dr. Horace Brown, F.R.S., in the latter's classic researches, and then and subsequently his work in connection with the chemistry of brewing showed his great ability, energy, and power of research.

In addition to nine new ordinary members enrolled, we have had the pleasure of including among our honorary members Dr. T. E. Thorpe, F.R.S., the head of the Government Laboratory. Not alone for the recognition shown us do we welcome Dr. Thorpe, but also because, in his official position, he has already given us proofs of his desire to co-operate with us in our work, and to bring about a better understanding between Public Analysts and Government Departments. We may sincerely look forward to much good resulting, alike as regards ourselves and the public interest, from the more ready interchange of views on analytical methods and practices which has characterized Dr. Thorpe's tenure of his high official post, in the more frequent association of leading members of our Society in public inquiries, and in the holding, from time to time, of conferences to settle debatable points, as was the case with the recent one on the margarine question.

The finances of the Society, thanks largely to our Honorary Treasurer, continue in a satisfactory condition, but it is necessary to remind members that there is need of a larger accession of new members to fill the place of those removed, to bear the considerable expense attaching to the issue of the ANALYST, and to provide for the useful development of its work. The ANALYST has well maintained the position it has gained, and it is now more than ever recognised as the journal to which one would naturally refer on points of new analytical methods or the examination of new and special materials. The work of abstracting from other sources has been kept well up to date, and to the Editor, Dr. Sykes, for his judgment and unremitting attention to make the ANALYST a worthy and useful journal, the chief credit is due. As a member myself, for the first time this past year, of the Editorial Committee, I can speak to the great trouble taken by almost each member of that Committee in revising the proofs, in making suggestions in and revisions of the abstracts, etc., and I have frequently been greatly struck by the accumulation of valuable experience and knowledge brought to bear on the subject-matter of the different issues as they come before the committee.

The list of papers read before the Society at its different meetings during the past year is as follows:

February 5.—"On the Authority of the British Pharmacopœia as a Standard under the Sale of Food and Drugs Act." By Alfred H. Allen.

"The Physical State in which Fat exists in Cream." By H. Droop Richmond and Sylvester O. Richmond.

"On the Composition of Dutch Butter." By John Clark, Ph.D.

"Note on the Proximate Analysis of Cloves." By A. McGill, B.A.

March 6.—"The Determination of Dissolved Oxygen in Water in Presence of Nitrites and Organic Matter." By S. Rideal, D.Sc.

"Some Analyses of Oatmeal." By Bernard Dyer, D.Sc.

"The Detection and Estimation of Preservatives in Milk." By M. Wynter Blyth, B.A., B.Sc.
April 3.—"On the Maumené Test for Oils." By C. A. Mitchell, B.A.
"Some Arsenic Estimations relating to Malt Kilns." By Thomas Fairley.
"On Gutzeit's Test for Arsenic." By F. C. J. Bird.
"The Aëration Test for Effluents." By S. Rideal, D.Sc.
May 1.—"Alkaline Waters from the Chalk." By W. W. Fisher, M.A.
"Citron Oil." By Herbert E. Burgess.
"Note on the Reduction of Iron Salts." By Gilbert T. Morgan, D.Sc.
June 5.—"Arsenic in Coal and Coke." By Alfred C. Chapman.
"Public Analysts' Records." By J. F. Liverseege.
"The Use of Partially Sterilized Milk Cultures in judging the Purity of Water." By H. Droop Richmond.
November 6.—"The Composition of Milk." By H. Droop Richmond.
"The Determination of Carbon in Steel by Direct Combustion." By Bertram Blount.
"*Enteritidis Sporogenes* as Evidence of Sewage Pollution." By M. Dechan.
December 11.—"A Contribution to a Knowledge of the Chemistry of Cider." By Alfred H. Allen.
"On the Mannitic Fermentation of Wine." By Philip Schidrowitz, Ph.D.
"On Cocoa Extracts." By E. G. Clayton.
"Note on Commercial Zinc White and other Pigments." By E. G. Clayton.

A glance at this list will show the varied nature of the subjects dealt with, and, while a large number of them have been, of course, concerned mainly with the work of the Public Analyst in relation to the Food and Drugs Act, yet a considerable number of them have been on subjects of wider application. Such, for instance, were the exhaustive paper by our late President, Mr. Fisher, on Alkaline Waters from the Chalk; that on the Chemistry of Cider by Mr. Allen; two papers by Dr. Rideal on the Determination of Dissolved Oxygen in Waters and on Aëration of Effluents; and, not least important, the interesting paper by Mr. Blount on the Direct Estimation of Carbon in Steel. Nor were matters of special interest to Public Analysts neglected, for we had a useful discussion introduced by a paper from Mr. Allen on the Value of the British Pharmacopœia as a Standard; the important and then burning question of the presence and determination of arsenic in foods and fuels was brought up; and, lastly, Mr. Richmond gave his annual résumé of his work of the year as concerns milk, but supplementing it with many matters of interest which travelled far beyond the merely statistical area.

As regards the meetings themselves, it is with pleasure that an improved and increasing attendance at the ordinary gatherings can be chronicled, and a further satisfactory feature has been the interest evoked in the discussions which have followed the reading of papers, and which has done much to make these latter profitable. The more numerous attendance has been due in no small measure to a happy inspiration of our Editor, Dr. Sykes, who, by the formation of a Dinner Club, which meets after the usual Council meeting is over, and before the evening meeting, brings the members together in friendly company, and contributes greatly to the promotion of kindly feeling and to better acquaintance of one member with another. The Council would take the opportunity of letting this be better known, and that all members of the Society can, at a small cost to cover postage, become members of the Dinner Club, and will be regularly informed of place and time of meeting.

The Annual Dinner of last year, which had been fixed for February 5, was, owing to the national mourning following on the deeply-lamented death of Queen Victoria, abandoned for the year. It is now fixed for February 12, and I am happy to say that we shall be honoured then by the presence of Mr. Hanbury, the President of the Board of Agriculture, the Secretary of the Board, and other distinguished guests.

And now it is fitting that I should review the principal events of the past year so far as these concern the position of our Society and its members in their public relations.

The only completed act of legislation which has affected us as Public Analysts is the framing of regulations by the Board of Agriculture in respect of milk. But other matters which have been under consideration, and will probably shortly have effect given to them, have seriously concerned us, and to them reference must also be made. These are the report of the Committee on Preservatives and Colouring Matters in Articles of Food, the inquiry into the cases of poisoning at Manchester through arsenic found in brewing materials, the Departmental Committee of the Board of Agriculture on Butter, and the Poisons Committee of the Privy Council.

Taking first the regulations with regard to milk, it will be remembered that a Departmental Committee was appointed early in 1900 by the Board of Agriculture to draw up regulations which should govern—in respect of their quality—the sale of milk and cream. Of this Committee Lord Wenlock was the chairman. Though, somewhat strangely, not numbering among the members a single Public Analyst in the strict sense, the Departmental Committee availed themselves very largely of the evidence and experience of Public Analysts and others belonging to our Society, and, it may be added, gave, in their report, great weight to the views of these representatives and to the conclusions adopted, with general agreement, by the Society for the guidance of its members. Ultimately the report of the Departmental Committee, issued in March, 1901, recommended a governing limit for milk of 12 per cent. of total solids, and that if a milk failed to give that quantity it should be further inquired into; that if then it be found to have less than 3·25 per cent. of fat, or to have less than 8·5 per cent. of non-fatty solids, a presumption should be raised, until the contrary was proved, that it was not genuine. In regard to cream, the Committee did not advise the fixing of any definite limits, but wished to prohibit the artificial thickening of it. Skimmed and separated milk were to have at least 9 per cent. of total solids, and condensed milk not less than 10 per cent. of fat and 25 per cent. of non-fatty solids.

The President of the Board of Agriculture did not see his way to adopt the limits which the Departmental Committee had advised, and finally, in August, 1901, regulations were issued by the Board of Agriculture, requiring milk, in order to be considered genuine, to contain not less than 3 per cent. of fat and not less than 8·5 per cent. of non-fatty solids. Skimmed and separated milk are in future to contain not less than 9 per cent. of total solids, but no regulations are made applying to cream or condensed milk.

That such result can be wholly satisfactory to those of us who have experience of the working of the Act is more than can be expected, and there must be a general feeling that the limits might very well have been extended, as proposed by the Departmental Committee, and a great deal of adulteration of milk been prevented, while, at most, the fixing of the higher limits would only have raised a "presumption" of want of genuineness, leaving further inquiry to be made. The opportunity, we think, might well have been taken to strike a blow at systematic "toning down" of milk, and to have given the country a higher quality of milk. Still, there is no getting away from the fact that the proposal to set the limits higher frightened, as we believe unnecessarily, the producers of milk—*i.e.*, the farmers—and they were hardly prepared for so marked a change as was proposed, and in deference to their views the Board of Agriculture declined to at once adopt the higher figures. Something, too, has now been gained in a definite pronouncement that milk that is sold shall have at least 3 per cent. of fat. The absence of regulations as to condensed milk is, however, to be regretted, for there can be little doubt that it is in the sale of condensed milk, deprived to a large extent of its nutritive qualities, and with the deficiency made up by the unrestricted addition of sugar, that great injury is done to infant life, especially in the poorer districts of large towns.

Another matter of kindred importance is the inquiry of the Departmental Committee of the Board of Agriculture into the regulations that shall guide the sale of butter. The Society of Public Analysts is represented on this Committee by one of its former Presidents. The less intricate part of the inquiry—that concerning the amount of water allowable in butter—has already been dealt with; but the more complex one—the composition of the fats and their determination—remains; and this, in view of the continual recurrence of what may be called "abnormal" butters, will inevitably present much difficulty. It is very clear that we know comparatively little about butter, and that there is much that has still to be made the subject of patient research and practical inquiry before we can properly formulate any clear distinction between what is butter and what is not. The Society of Public Analysts was asked to express its views through two of its representatives, and Dr. Dyer and Mr. Hehner have tendered their evidence on behalf of the Society.

Yet another Departmental Committee, that on Food Preservatives, appointed by Mr. Chaplin in 1899, has been conducting an inquiry in which analysts are much concerned, and has just reported its recommendations. The evidence of several prominent members of our Society was given before this Committee, and the recommendations arrived at, it may be said generally, meet with the approval of this Society. Special satisfaction will be felt at the recommendation which advocates the entire prohibition of the use of preservatives or colouring matter in milk. Next to this in importance is the suggested prohibition of formaldehyde and the limiting of the amount of boracic preservatives in cream, butter, and margarine. It is sincerely to be hoped that these recommendations of the Committee will be carried into effect.

The serious outbreak of illness in Manchester and district towards the close of 1900, and which was traced to the presence in beer, through the brewing materials used, of arsenic, caused great concern and anxiety to Public Analysts throughout the country, and for some months, it may be said, the laboratories of many members of our Society were turned into beer depots and arsenic-testing establishments. Needless to say, the work was of a most delicate nature, and on the Analysts' reports hung important issues. As an outcome of the spread of the epidemic, and the serious results that in many cases followed, a Royal Commission was appointed to inquire into the whole matter; and here, again, members of this Society rendered great service by the evidence they tendered, and still more by the immense amount of work which they did in the effort to arrive at definite conclusions as to the occurrence of arsenic in different materials used for brewing, and in other articles of food and drink, and as to the definite quantitative estimation of small amounts of arsenic in such materials. The papers read before this and kindred societies testify to the vast amount of work done by members of the Society to arrive at the truth, and this culminated in the appointment, jointly by the Society of Public Analysts and the Society of Chemical Industry, of a Committee to examine and report upon the best methods for the estimation of arsenic in minute quantities. This Committee has just recently presented its report, which has been brought before both Societies and will shortly be published. Meanwhile the best thanks of this Society are due to those of its members who were on the Committee, and they may be congratulated on the careful and exhaustive nature of their work, and on the unanimous conclusions come to. It has now been announced that the Royal Commission, above alluded to, has appointed a Committee to conduct on their behalf an inquiry into, and formulate methods for, the determination of arsenic in food materials. Among the experts named are Dr. Thorpe and Professor Tilden. I think we cannot do better than refer the Committee to the report of the Joint Committee of the Society of Chemical Industry and Society of Public Analysts.

During the past year several new forms of adulteration have come to the front, new forms of food articles have been introduced, and legal decisions have been given, all of which affect the position of Public Analysts, and to which brief reference may be made. Perhaps the new material to which most attention has been drawn is that known now as "Milk-blended butter," but sold at first simply as "butter." This article is made by taking colonial butter and churning it up with milk, preservatives being added to prevent the milk "souring." The product contains from 20 to 25 per cent. of water, instead of the 16 per cent. which analysts generally agree should be an extreme limit for butter. By a decision of the High Court of Justice, in August, 1901, this material could no longer be sold as "butter," and the term "milk-blended" was thereupon ingeniously introduced to allow of the material being sold, as, if not sold as butter, it could not, by the provisions of the Margarine Act, be offered as margarine, the proportion of butter in it exceeding the allowed 10 per cent. Hence the importance to the sellers. Many have been the proceedings taken against this article, and various have been the decisions given, and the point at issue cannot really be satisfactorily settled without a High Court decision being given upon it. It cannot, however, but be clear to anyone who has knowledge of the article that the incorporation of milk with butter is done purely with the object of making, as Mr. Justice Wills said, "a spurious profit," and it is equally clear that the qualifying term "milk-blended" is a misleading one, and does not convey to the public the idea of the true nature of the article, or that it is butter which has been reduced in value and composition by the addition of milk and preservatives to it. One may very rightly ask where the mixing is to stop; for if 10 or 20 per cent. of milk may now be added to butter, and the mixture be sold by a name not disclosing its real nature, what is to prevent 30, 40, or even more, per cent. being added if there be found means of incorporating it?

The same point has quite recently been raised with regard to the latest form of adulteration—the addition of water to margarine. If butter may not have more than 16 per cent. of water, why should margarine be allowed to have more? and if it be maintained that there is no limit for water in margarine, where is the addition to stop? Why may not 30 or 40, or more, per cent. of water be put in if 20 per cent. is allowed? The necessity of restriction in this direction is clearly apparent.

The difficulties of the butter question have already been alluded to; but just as the analyst was beginning to think that he was coming to some understanding about butter and the discrimination of butter-fats and animal and vegetable fats and oils, the introduction, in large quantities, of butter from Siberia, made under conditions of climate, temperature, etc., vastly different to those of our own country, has brought in further complications, for this butter has been found to give results, as regards the volatile and non-volatile fatty acids, of quite abnormal nature. The question thus opened up is a very serious one. It is now recognised that butter made under exceptional conditions, as, for instance, in a very cold climate, and when cows are kept out in the open longer than is usual, will give analytical results which do not conform to those of butter as ordinarily produced; and though it may be nothing but the product of the churned cream of the milk of the cow, it may yet approach in composition to margarine or other fats. Inasmuch, therefore, as the analyst has not the means of knowing where his sample comes from, the discrimination of margarine and foreign fats from genuine butter is rendered still more difficult. It remains then, it would seem, a question of public policy whether such "abnormal" butters should be allowed to enter and be sold as butter, or whether, as was done in the case of the Margarine Act, exclusion of certain kinds should not be insisted on and limits be fixed to which all genuine butter should conform. I must not pursue this matter further here, but leave the rest to the consideration of the Departmental Committee on butter regulations, who, however, I anticipate, will find their task no easy one.

The last form of adulteration to which I shall refer is that of glucose in marmalade. After the obtaining of a conviction in the police-court at Worthing, and the upholding of the conviction at the Horsham Quarter Sessions, for the sale of marmalade to which glucose had been added, the same not being declared, the case was taken on appeal to the High Court, King's Bench Division. The Lord Chief Justice, in giving the decision of the Court, quashed the conviction on the ground that glucose was not found to be injurious to health, and that, as the object of the Sale of Food and Drugs Act was the protection of health, it ought not to be construed as an instrument of oppression. It was allowed that a different article had been supplied to that asked for, but the Justices held that the article was not an inferior, but possibly rather better one, and that there was no evidence of inferior quality or adulteration in the ordinary sense of the word.

Though it is not for me to discuss the legal bearings of the case, I am sure all Analysts will agree with me that the decision of the Lord Chief Justice gives a quite different interpretation to the Act to any we have ever associated with it; and if the test to be applied in the case of alleged admixture without declaration of the nature of the admixture is merely whether the material is injurious to health or whether it is superior or inferior to that asked for, there is no telling where admixture will cease. It would be quite easy to show that vegetable marrow and carrots were not injurious to health, and gave, when mixed with fruit, quite as good a jam as whole fruit; but, for all that, the material sold would not be of the "nature, substance, and quality demanded" by the purchaser. This decision, following on the important point gained in the case of golden syrup, whereby glucose, if used, must be declared to be present, seems to me a most unfortunate one so far as the public and the Public Analyst are concerned.

Other legal decisions that bear on the Public Analyst's position concern the sufficiency of the present form of certificate, and the non-obligatory nature of the instruction to insert the weight of the article sent for analysis.

In our relations to Government Departments and to outside bodies several matters of special interest have come to the fore. We cannot help seeing, and with some satisfaction, the tendency to bring our work into closer connection with the Board of Agriculture, in place of the Local Government Board, which latter body discharges duties, as affects the Public Health, more of a medical than a chemical character, and has relation, thus, rather to the Medical Officer of Health than to the Public Analyst. The desirability of separating, as far as possible, the work of the Medical Officer of Health and of the Public Analyst has been more and more apparent, and has formed the subject of strong representations to the Local Government Board both from the Institute of Chemistry and the Society of Public Analysts. Reference to the action taken is made in the ANALYST for December, 1901, and it is confidently believed that the representations made will not be without good effect.

One other matter I must briefly touch upon, as it is one that depends for remedy upon our members themselves, and on the growth and cultivation of a higher sense of professional status and its requirements. I refer to the acceptance by members of the Society of appointments under terms which are not worthy of the dignity or responsibilities of the office. So long as there are men who are willing to accept appointments under such conditions, one can hardly blame Corporations and Local Authorities for encouraging them to underbid one another. But this is not "cricket," as one might say, and we might join with Rudyard Kipling in bemoaning the decadence of a true spirit of duty and obligation.

I have endeavoured to show in my address that the work of the Society is going on well and steadily, and that more and more demand is being made upon its members to give the benefit of their experience and opinion on matters of public inquiry, Departmental Committees, and the like, and I feel sure that with hearty co-operation and the raising of the standards alike of efficiency and honour the analytical chemist and Public Analyst will become an increasingly useful and necessary individual. One word of warning I would interpose. There is just a danger that, with all the fixing of limits towards which legislation seems to tend, there will be a disposition (to which, indeed, expression has been occasionally given) to degrade the Public Analyst's position to that of a mere recorder—a machine, in fact, to turn out certain figures, and to leave these, without comment or interpretation of their significance and bearing, to be interpreted by others unacquainted, necessarily, with the real points at issue. It may be well to have limits defined, but the application of these must be governed by the judgment of those who have had the responsibility of making them. To regard the Public Analyst merely as one who has to supply certain figures and to have no further voice, if his authorities wish to consult him, is to altogether misunderstand his true position, and to surrender the work by which it has been gained, and I sincerely trust that members of this Society will be alive to this danger which possibly exists in the future, and will strongly resist any attempt to reduce their status to that of an analytical machine.

In conclusion I would express the pleasure it has given me to preside, however imperfectly, over your deliberations and proceedings this year, and to acknowledge, as I heartily do, the help given me by the past Presidents, members of the Council, and, not least, by the very efficient Secretaries of the Society, Mr. Bevan and Mr. Chapman.

SOME NOTES ON THE ANALYSIS OF DRUGS.

By Sir Charles A. Cameron, C.B., M.D.

Determination of Alcohol in the Tincture and Solution of Iodine (*B.P.*).—In the *Pharmaceutical Journal*, 1898, ii., 333, it is recommended to fix the iodine with potash before distilling the tincture or solution. I find that the potash often causes "bumping," and there is some danger that an excess might be used which would act upon the alcohol. The addition of iron turnings to the tincture rapidly fixes the iodine, and is an improvement on the potash method.

Syrup of Ferrous Phosphate with Quinine and Strychnine.—The small quantity of strychnine and the large amount of quinine in this syrup render the estimation of the former a difficult operation, especially as only small quantities of the syrup are usually sent for analysis. I find the following method satisfactory: Both alkaloids are precipitated together and weighed together. The sulphuric acid is then determined and calculated as sulphate of quinine; the excess of alkaloid is strychnine. In 100 c.c. of this syrup there are 14·8 grammes of quinine sulphate, and only 0·57 gramme of strychnine.

Tincture of Assafœtida.—In Gadd's useful little book, "A Synopsis of the Pharmacopœia," fifth edition, it is stated that tincture of assafœtida contains 10 per cent. of solid matter in solution. In specially-prepared tincture I find that the solid matter is only 5 grammes per 100 c.c. The Pharmacopœia states that assafœtida should dissolve to at least 62 per cent. in spirit of 90 per cent. alcohol. It would have been better if its solubility in spirit of 70 per cent. alcohol had been given, as that spirit is the one employed in the preparation of the tincture. As the tincture is prepared by acting upon 200 grammes of the gum with 1,000 c.c. of spirit of 70 per cent. alcohol, one might naturally think that the spirit would hold about 10 per cent. of solid matter in solution; but this, as I have stated, is not the case. When the tincture containing 5 grammes of solids per 100 c.c. is mixed with 5 per cent. of water, it becomes clouded.

Tincture of Nux Vomica.—In Gadd's book it is stated that this tincture contains 45 per cent. of alcohol, but if it is prepared according to the Pharmacopœia it should contain 52·2 per cent. of absolute alcohol by volume.

Solution of Ferric Chloride.—The Pharmacopœia gives the specific gravity of liquor ferri perchloridi as 1·11. It is prepared by diluting the strong solution of ferric chloride, and as its strength is stated to be the same as that of the tincture of ferric chloride, it should contain 5·625 grammes of iron per 100 c.c. I find, however, that unless the specific gravity is greater than 1·11 it does not contain the supposed amount of iron.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

Estimation of Fusel Oil in Brandy or other Alcoholic Liquors. E. Beckmann. (*Zeit. für Untersuch. der Nahr. und Genussmittel,* 1901, iv., 1059-1064.)—The following method was found by the author to give satisfactory results: 50 c.c. of the brandy, together with 20 grammes of pure granular calcium chloride, are placed in a separating funnel, and so much water is added that the alcohol in the solution shall not exceed 50 per cent. When the calcium chloride has dissolved, the solution is thoroughly well extracted with four successive quantities of about 30 c.c. each of carbon tetrachloride. These extractions are collected in another separating funnel, and shaken with 25 c.c. of water to remove ethyl alcohol. The water may then be shaken with 80 grammes of carbon tetrachloride, after the addition of 10 grammes of calcium chloride. The total amount of carbon tetrachloride is dried by means of a little fused calcium chloride, and filtered through a plug of glass wool into a stoppered flask, the glass wool being washed with a little dry carbon tetrachloride. Three grammes of powdered acid sodium sulphate and 3 grammes of sodium nitrite are then added, and the flask is repeatedly shaken for thirty minutes. The solution is again filtered through glass wool, and the saline residue washed with a little carbon tetrachloride. The filtrate is collected in a stoppered flask, and 3 grammes of powdered acid sodium carbonate are added. This removes the excess of nitrous acid. Water is added after the evolution of carbon dioxide has ceased, and the solution of the esters in the carbon tetrachloride is drawn off by means of a separating funnel. Ten c.c. of concentrated sulphuric acid are then added to decompose the esters and the mixture well shaken; 100 to 170 c.c. of ice-water containing pieces of ice are then added, and the solution is titrated with a solution of potassium permanganate (1 gramme per litre). Aldehydes, when present, interfere with the accuracy of the results, and may be removed by shaking the carbon tetrachloride solution with acid sodium sulphite and washing out the aldehyde compound formed with water. As this may introduce traces of sulphurous acid, the nitrite should be estimated in a nitrometer. W. P. S.

Detection of Heated Milk. M. Siegfeld. (*Milch Zeit.*, 1901, xxx., 723; through *Chem. Zeit. Rep.*, 1901, 366.)—The author has investigated Storch's *p*-phenylenediamine test for raw milk, and he finds that the temperature of heating which causes that reaction to fail is between 71° and 80° C., being chiefly dependent upon the acidity of the milk, and partly upon the time over which the heating extended. Therefore the test affords no method of discovering whether the milk has been raised to any temperature exceeding 80°. Still, it is so delicate that it will show the presence of 10 per cent. of raw milk in heated milk. The best method of carrying out the test is to shake about 10 c.c. of the sample with 1 or 2 drops of "medicinal" hydrogen peroxide, then adding 2 or 3 drops of a 2 per cent. alcoholic solution of *p*-phenylenediamine. A bluish colour will develop in the heated-milk tube on exposure to air and light, but the tint only becomes pronounced after several hours.

F. H. L.

Detection of Heated Milk. Du Roi and Koehler. (*Der Landbote*,* No. 103, 1901; through *Chem. Zeit. Rep.*, 1902, 13.)—Fifty c.c. of the milk are shaken with 1 c.c. of 1 per cent. hydrogen peroxide, and 3 c.c. of the mixture are poured into a tube containing an equal volume of a solution of starch and potassium iodide. If the milk is raw, thorough agitation causes the appearance of the blue iodine colour; but with heated milk the mixed liquids remain colourless. The test will show 2 per cent. of raw milk in heated milk. Potassium bichromate and formaldehyde, used in the ordinary proportions as preservatives, do not interfere. Raw sour milk yields the blue colour immediately. The critical temperature is about 80° C.

F. H. L.

Notes on the Estimation of Phosphoric Acid in Milk, etc. F. Rieger. (*Zeits. physiol. Chem.*, 1901, xxxiv., 109; through *Chem. Zeit. Rep.*, 1901, 366.)—In order to obtain an ash perfectly free from carbon, 50 c.c. of the milk should be evaporated to a syrup in a large platinum basin, stirring continuously, and finally adding some dry, powdered sodium carbonate. The residue is carbonized and ignited for fifteen minutes, and then heated once again, with agitation, under a layer of 1 part of sodium carbonate and 2 of potassium nitrate, until it becomes pasty. The product will be found to dissolve in cold nitric acid to an almost absolutely clear liquid.

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* This is the organ of the Landwirthschaftskammer for the province of Brandenburg.

For the estimation of total phosphorus in milk the sample may be precipitated with sodium hydroxide and copper sulphate solution, as in Ritthausen's process for the determination of albuminoids. The precipitate, which contains all the phosphorus as well as the albuminoids, is then incinerated with sodium carbonate and potassium nitrate as above described.
F. H. L.

So-called Honey Dextrin. **E. Beckmann.** (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1901, iv., 1065-1069.)—When 5 c.c. of a 20 per cent. solution of pure coniferous honey are mixed with 3 c.c. of a 2 per cent. solution of barium hydroxide and 17 c.c. of methyl alcohol, little, if any, precipitate is obtained. Should the honey, however, contain starch syrup, a precipitate is formed. This shows that the dextrin of coniferous honey is different to that present in starch syrup. The author has investigated the behaviour of the different dextrins of commerce, and finds that they give the following barium precipitates when treated as above:

A 20 per cent. Solution of:	Colour given with Iodine Solution.	Barium Precipitate.
Dextrin puriss. Merck	Cherry red	104·9 per cent.
,, technic. Merck	Dark blue	91·5 ,,
,, granular. Merck	Cherry red	86·45 ,,
,, Pharm. Germ. I. Merck ...	,, ,,	78·4 ,,
,, from starch syrup	No colour	41·3 ,,
,, from starch sugar	,,	29·3 ,,
,, from honey	,,	8·18 ,,

E. Prior found that achroodextrin III., giving 44 per cent. of barium precipitate, had a molecular weight of 642, and the formula $2(C_{12}H_{20}O_{10}) + H_2O$. Honey dextrin has apparently a still lower molecular weight.

Similar results to the above were obtained by using basic lead acetate in place of barium hydroxide, the yield of precipitate being somewhat larger with the former. The sugars in honey to some extent prevent the precipitation of dextrin, so that small quantities of added starch syrup may not be detected by this test.

J. Monheim ascertained that the benzoyl esters of honey dextrin correspond to a mixture of penta- and hexabenzoate of a disaccharide. Like the benzoates of saccharose and maltose, these compounds of honey dextrin are soluble in ether, whilst the benzoate of ordinary starch dextrin was almost insoluble. The benzol sulphonic esters obtained from honey dextrin corresponded with a disaccharide with 4 to 6 acid radicals, the *m*-nitro-benzoate to a saccharide with 5 to 6 acid radicals.
W. P. S.

The Detection of Artificial Colouring Matters in Sausages. E. Spaeth. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1901, iv., 1020-1023.)—The finely-divided sausage is heated for some hours at 100° C. and then extracted with ether. The fat-free substance is now warmed on the water-bath with a 5 per cent. solution of sodium salicylate for one hour. The coloured solution (if colouring matter be present) is filtered, acidified with sulphuric acid, and heated with some wool free from fat. It is not necessary to remove the precipitated salicylic acid by means of ether, etc., as it re-dissolves on warming, and in no way interferes with the fixing of the colour by the wool. The dyed wool may then be used for the identification of the colour. W. P. S.

The Adulteration of Pepper with the Fruit of Myrsine Africana and Embelia Ribes. A. Mennechet. (*Journ. Pharm. Chim.*, 1901, xiv., 557-561.)—The adulteration of pepper with these substances is still practised, though, according to the author, less frequently since 1889.

After giving a description of the structure of the two fruits, as seen under the microscope, the author points out that it would not be easy to identify a small addition of either of them to pepper by this means, and he therefore recommends the following chemical test: The powder is lixiviated with ether, and the yellow extract shaken with several times its volume of water rendered alkaline with ammonia. The lilac-red colouring matter (ammonium embelate) formed is insoluble in ether, but is dissolved by the water. On the addition of an acid the red colour disappears, and the embelic acid again dissolves in the ether.

Myrsine, which gives the same reactions, appears to contain a closely-related compound.

No coloration is given by pure pepper when thus treated. C. A. M.

The Official Test for Myrrh. H. G. Greenish. (*Pharm. Journ.*, 1901, 666.)—As an improvement on the official test, the following is recommended: Half a gramme of coarsely-powdered myrrh, occasionally shaken during ten minutes, with 10 c.c. of ether, should afford a filtrate, 2 c.c. of which should yield, when evaporated, a residue that is slowly coloured violet by contact with the vapour of nitric acid. Bissabol, bdellium, hotai, and other gums, do not give any violet reaction.

W. P. S.

Notes on the Estimation of Berberine. H. M. Gordin. (*Amer. Journ. Pharm.*, 1902, lxxiv., 37-39.)—In the author's first method of determining berberine (*Arch. d. Pharm.*, 1901, 638; *cf.* ANALYST, xxiv., 238), the alkaloid is extracted from the drug by means of hot alcohol, the solution made up to a definite volume with cold alcohol, and the berberine precipitated as an acid sulphate which is subsequently converted into the mono-acid hydriodide.

Recent experiments have shown that although berberine in the form in which it occurs in *Hydrastis canadensis* and some other plants is readily soluble in alcohol, yet in others, such as barberry bark, it is frequently present in a form which only dissolves with difficulty in cold alcohol; so that on adding cold alcohol to the hot alcoholic extract some of the berberine is precipitated.

To guard against this the sediment should be dissolved in water, filtered, and the clear filtrate tested for berberine. If present, it should be determined by the second method described in the former paper (*loc. cit.*).

As regards the precipitation of berberine by potassium iodide, the author states that in the presence of free acid Mayer's or Wagner's reagents effect more complete precipitation, and it is therefore advisable to use Mayer's reagent in the first method, in which acid is liberated in the reaction.

A further improvement is to collect the berberine acid sulphate upon a plug of cotton-wool in a filter, and to wash the beaker out twice with 5 c.c. of a mixture of alcohol and ether (1 : 1), and then to wash the precipitate with ether, in which it is completely insoluble. The amount of berberine in the 10 c.c. of alcohol-ether washing is less than 0·0006 gramme, and may be neglected. C. A. M.

TOXICOLOGICAL ANALYSIS.

The Detection of Free Phosphorus in the Presence of Alcohol. J. Habermann and A. Oesterreicher. (*Zeit. anal. Chem.*, 1901, xl., 761-766.)—Various substances, and in particular alcohol, interfere with the production of the phosphorescence in Mitscherlich's method of detecting phosphorus (*cf.* ANALYST, xxiii., 39). According to the results of experiments made by the authors, this difficulty is avoided by the dilution of the alcoholic vapour or distillate with a sufficient quantity of water; and on this fact are based the following modifications of Mitscherlich's process:

1. The liquid is distilled in a dark room, and as soon as the steam is felt by the hand to have reached the upper part of the condenser, water is introduced through a stopcock.
2. The liquid is fractionally distilled in daylight, and the several fractions diluted with water in the dark. C. A. M.

Detection of Phosphorus in Cases of Poisoning. C. Binda. (*Giorn. Farm. Trieste*, 1901, vi., 225; through *Chem. Zeit. Rep.*, 1901, 354.)—It is known that certain liquids, like ethyl alcohol and turpentine, prevent the vapours of phosphorus from being visible in the dark. In such cases the author finds luminosity to occur if the liquid is evaporated rapidly on a glass plate in the dark, the plate being rubbed with a glass rod meanwhile. F. H. L.

The Alleged Poisonous Properties of the Colours "Mandarin" and "Metanil Yellow." J. Frentzel. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1901, iv., 968-974.)—In this paper are given the results of a considerable number of experiments, consisting in feeding rabbits, dogs, and human beings with food mixed with the colours "mandarin" (obtained by diazotising sulphanilic acid and β-naphthol) and "metanil yellow" (prepared by diazotising meta-benzene-sulphonic acid and diphenylamine). The conclusions arrived at are that the colours can scarcely be considered poisonous in the small quantities in which they are used in foods, etc. Long-continued, large doses, however, give rise to some injurious effects; but this quantity is never, in the natural course of things, even approximately reached. W. P. S.

ORGANIC ANALYSIS.

Gravimetric Determination of Formaldehyde. L. Vanino. (*Zeit. anal. Chem.*, 1901, xl., 720, 721.)—This method is based upon the facts that, on treating a solution of formaldehyde with silver nitrate and sodium hydroxide, a mixture of metallic silver and silver oxide separates, and that the silver oxide is dissolved on adding dilute acetic acid to the mixture. Thus:

$$4AgNO_3 + 4NaOH = 2Ag_2O + 4NaNO_3 + 2H_2O$$
$$2Ag_2O + 2NaOH + 2HCOH = 2HCOONa + 4Ag + 2H_2O.$$

In making a determination an aqueous solution of 2 grammes of silver nitrate is rendered strongly alkaline with sodium hydroxide, 5 c.c. of a 10 per cent. solution of commercial formalin introduced with continual stirring, and the whole put aside in a place protected from the light.

After fifteen minutes the clear supernatant liquid is poured off through a weighed filter and the precipitate digested three or four times with 5 per cent. acetic acid, transferred to the same filter, washed with water rendered slightly acid with acetic acid until free from chlorine, dried at 105° C. and weighed.

The results quoted are very concordant and in close agreement with those obtained by other methods. C. A. M.

On the Separation of Terpene Alcohols from their Esters by means of Sodium Salicylate. G. Darzens and P. Armingeat. (*Bull. Soc. Chim.*, 1901, xxv., 1053-1055.)—Charabot and Hébert have recently described a method of separating terpene alcohols from their esters by treating the mixture with a 50 per cent. solution of sodium salicylate, in which only the alcohols were said to be soluble. (*Bull. scient. et indust. de la Maison Roure-Bertrand*, October 5, 1901; *cf.* ANALYST, xxv., 72, 188.)

The authors have made experiments with this method, and have come to the conclusion that little reliance can be placed upon it as a quantitative method. Mixtures of linalol, geraniol, and rhodinol with their respective esters were prepared and the saponification values of the mixtures determined. They were then treated four times with an equal volume of 50 per cent. sodium salicylate solution, and after standing for twenty-four hours the insoluble portion was decanted and treated with water in excess. Finally, the saponification value of each of these fractions was determined.

The following table of results thus obtained shows that in each case the dissolved portion contained considerable quantities of esters.

MIXTURE.			INSOLUBLE PORTION.		SOLUBLE PORTION.	
	Saponification Value.	Esters per Cent.	Saponification Value.	Esters per Cent.	Saponification Value.	Esters per Cent.
Geraniol and geranyl acetate	116	40·6	110	38·5	120·8	42·3
Linalol and linalyl acetate	98·2	34·4	116·3	40·7	85	29
Rhodinol and rhodinyl acetate	126	45	Mixture completely soluble			

C. A. M.

A New Colour Reaction for Cholesterol. **L. Tschugaew.** (*Jour. russ. phys.-chem. Gesellsch.*, 1900, xxxii., 363; through *Zeit. für Untersuch. der Nahr. und Genussmittel*, 1901, iv., 974, 975.)—The test consists in dissolving cholesterol in glacial acetic acid, and adding an excess of acetyl chloride together with a small piece of zinc chloride. On warming, a red or rose coloration is produced, which also shows a greenish-yellow fluorescence. The maximum depth of colour is reached after five minutes' boiling. The test will detect 1 : 80,000. W. P. S.

The Detection of Vegetable Oils in Animal Fats by Means of the Phytosterol Acetate Test. **A. Bömer.** (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1901, iv., 1070-1095.)—The method of carrying out this test is as follows: From 100 grammes of fat the crude cholesterol and phytosterol are obtained in the usual manner by shaking out the saponified fat with ether. The ethereal solution is well washed with water to remove dissolved soap, and the greater part of the ether is distilled off. The remainder of the ether is then allowed to completely evaporate in a small basin; the residue is taken up in the least possible quantity of alcohol, and allowed to crystallize. These crystals may be examined microscopically to see whether cholesterol or phytosterol be present. The alcohol is then completely driven off on the water-bath, 2 to 3 c.c. of acetic anhydride are added, and the contents of the basin boiled for a quarter of a minute over a wire-gauze, the basin being covered with a clock-glass. The excess of acetic anhydride is evaporated on the water-bath. Sufficient absolute alcohol is now added to just dissolve the esters on heating. To prevent immediate crystallization, 1 c.c. more of absolute alcohol is added, and the esters allowed to crystallize at the ordinary temperature of the room. After one-half to one-third of the alcohol has evaporated and the greater part of the esters have crystallized out, the crystals are filtered through a small filter, again dissolved in alcohol, and allowed to crystallize. This recrystallization is repeated as many times as the quantity of the crystals will allow. The melting-point of the crystals is taken after the third and subsequent crystallizations. Should the melting-point of the crystals be 116° C., the presence of vegetable oil may be suspected; if the melting-point be 117° C., or higher than this, vegetable oil is certainly present. The melting-point should be corrected by the formula $S = T + n(T - t) \times 0{\cdot}000154$, where S is the correct melting-point, T the observed melting-point, n the length of the column of mercury above the surface of the liquid, and t the average temperature of the column of mercury outside the liquid. This latter figure is obtained by means of a second thermometer placed at the middle of the column. W. P. S.

Note on Halphen's Test for Cotton-seed Oil. P. Soltsien. (*Zeit. öffentl. Chem.*, 1901, vii., 140-143; through *Zeit. für Untersuch. der Nahr. und Genussmittel*, 1901, iv., 979.)—The author draws attention to the fact that American lards obtained from pigs fed on cotton-seed meal show strong reactions with Halphen's and Bechi's tests. The same lards, however, give no reaction with Welman's test, and phytosterin cannot be detected in them. The presence of added cotton-seed oil in lard is, therefore, only to be detected with certainty when confirmed by the two latter tests.

W. P. S.

Formula for the Calculation of Diglycerides in Fat Mixtures which contain Oxy-fatty Acids. J. Freundlich. (*Chem. Zeit.*, 1901, xxv., 1129.)—The formula given by Benedikt for this purpose is inconvenient, as it requires two acetylations—that of the original fat and that of the fatty acids. Calling the ether value of the fat d, the yield of total fatty acids F, and the molecular weight of the diglycerides M, their percentage amount is given by the formula

$$D = M \times \frac{1683(100 - F) - 38d}{51612}.$$

If no soluble fatty acids are present, F may be replaced by the Hehner number; otherwise that figure must be ascertained analytically. Presence or absence of oxy-fatty acids is immaterial, and they may exist either as neutral fats or in the free state. The steps involved in arriving at the above formula are explained in the original article. Freundlich remarks that both in the *Zeits. angew. Chem.*, 1888, 461, and in Benedikt's book (3rd edition, p. 165), there is an error in the corresponding formula quoted, the first figure in the denominator being given as "5600," instead of "56100." F. H. L.

Detection of Gallic Acid in Tanning Materials. M. Spica. (*Gazz. Chim. Ital.*, 1901, xxxi. [2], 201; through *Chem. Zeit. Rep.*, 1902, 4.)—The reagent is a solution of potassium plumbite prepared by dissolving in the necessary quantity (not an excess) of potassium hydroxide the precipitate obtained therewith from a solution of lead acetate. On adding some of this liquid to a dilute solution of gallic acid, shaking repeatedly so as to encourage oxidation, and finally diluting with water, a scarlet-red colour will be produced—a reaction which is neither given nor hindered by the presence of tannin. The method is capable of being made quantitative by colorimetry. F. H. L.

The Adulteration of Shell-lac. K. Dieterich. (*Chem. Revue ü. Fett und Harz-Ind.*, 1901, viii., 222-226 and 244-246.)—The results of a number of experiments with pure shell-lacs and mixtures of shell-lac and colophony lead to the conclusion that the determinations of the acid, ester, and saponification values are of little use for the detection of adulteration. The values obtained with mixtures still lay within the wide limits yielded by pure shell-lacs. Better results were obtained by determining the solubility of the samples in ordinary solvents, such as ether, petroleum spirit, benzol, etc. In ether colophony is insoluble, whilst shell-lac dissolves to the extent of 14 per cent. W. P. S.

Colour Reactions of Certain Coal-Tar Derivatives and Morphine with Formaldehyde and Sulphuric Acid. R. A. Hatcher. (*Amer. Journ. Pharm.*, 1902, lxxiv., 35, 36.)—The author's experiments have led him to the conclusion that the formaldehyde sulphuric acid test for morphine is a reliable one; but, at the same time, care should be taken to distinguish the colour from the colorations given by the other compounds mentioned.

It was found that, in the case of mixtures of phenol and morphine, a large proportion of the former could be expelled, without decomposing the alkaloid, by evaporating an alkaline solution with strong sulphuric acid.

In the tests tabulated below 1 drop of the respective solutions was added to 5 drops of the reagent prepared by mixing 5 c.c. of formaldehyde (40 per cent.) with 100 c.c. of sulphuric acid, the following results being obtained:

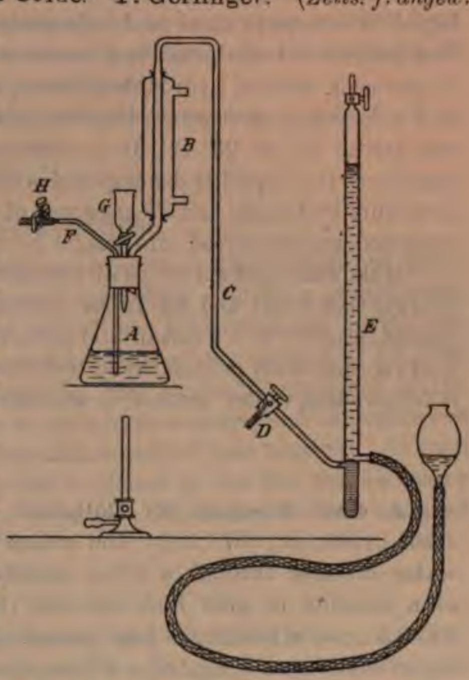
Phenol, 0·1 gramme + 5 c.c. $\frac{N}{10}$ NaOH, warmed and mixed with 10 c.c. $\frac{N}{10}$ H_2SO_4.	Morphine Sulphate, 0·01 gramme +5 c.c. $\frac{N}{10}$ NaOH +10 c.c. $\frac{N}{10}$ H_2SO_4.	Phenol, 0·1 gramme; Morphine Sulphate, 0·01 gramme+5 c.c. $\frac{N}{10}$ NaOH+10 c.c. $\frac{N}{10}$ H_2SO_4.	Sodium Salicylate (commercial), 0·1 gramme+50 c.c. $\frac{N}{10}$ H_2SO_4.	Sodium Salicylate (Chem. Pure).	Salicylic Acid (Chem. Pure) dissolved in Alcohol.
Cold.	*Cold.*	*Cold.*	*Cold.*	*Cold.*	*Cold.*
Crimson to cherry, deepening to garnet on standing.	Maroon, deepening on standing.	Deep crimson, inclining to garnet.	*Faint* pinkish salmon.	None.	None.
On Heating.	*On Heating.*	*On Heating.*	*On Heating.*	*On Heating.*	*On Heating.*
Maroon precipitate.	Fades to dirty brown.	Brown, maroon tint.	Rose-pink.	None, except fleeting rose-pink on superheated edges of vessel.	Same as sodium salicylate. Odour of oil of wintergreen.

It is advisable, in the presence of these substances, to remove them from the solution by extraction with ether after acidification, and to apply the test for morphine to the residual solution. C. A. M.

Gasometric Estimation of Nitrites in Urine. P. Gerlinger. (*Zeits. f. angew. Chem.*, xiv., 1250.)—The nitrite by double decomposition with ammonium chloride is converted into ammonium nitrite, which is decomposed by boiling according to the equation:

$$NH_4NO_2 = 2H_2O + N_2.$$

In the flask *A* is placed a saturated neutral solution of ammonium chloride which has previously been boiled. Carbon dioxide is led into the apparatus through the tube *F* to entirely displace air, the liquid being meanwhile heated. The flame is now removed. The cocks *D* and *H* are closed, and the urine or other substance to be examined is introduced carefully through the funnel *G*. The liquid is now gently boiled, and all the nitrogen is driven by means of a current of carbon dioxide into the nitrometer *E*, and measured in the usual way. The author finds that the results are accurate. A. M.



Jolles' Process for the Estimation of Uric Acid in Urine. O. Makowka. (*Chem. Zeit.*, 1901, xxv., 1159.)—The present author has investigated this process, and finds it to be convenient and exact, while its simplicity, in comparison with the gravimetric method or the volumetric process of Hopkins and Folin, renders it preferable for practical purposes. Jolles' process is best carried out as follows: 100 c.c. of urine, previously warmed to 60° C. and filtered if turbid, are mixed with 10 grammes of solid ammonium acetate (or sulphate) in a 600 c.c. flask, and from 3 to 5 c.c. of strong ammonia are added with constant stirring till the odour is persistent. The whole is allowed to rest for four hours, the clear liquid run off through a filter, and the deposit of phosphates and uric acid washed by decantation some nine times with 10 c.c. of 10 per cent. ammonium carbonate solution till the filtrate no longer gives a chlorine reaction. The precipitate is next rinsed into a beaker with water at 80°, the liquid adjusted to 50 or 70 c.c., and the mixture boiled for about thirty or forty-five minutes with 0·1 to 0·2 gramme of pure magnesia until litmus-paper shows that all ammonium salts have been decomposed. It is then acidulated with weak sulphuric acid, diluted to 200 or 300 c.c., and treated with 10 c.c. of 50 per cent. sulphuric acid (specific gravity 1·4). The whole is gently boiled, and a solution of potassium permanganate (8 grammes per litre) is run in, 1 c.c. at a time at first, afterwards in quantities of 6 drops, till the pink colour withstands fifteen or twenty minutes' boiling, the volume of the liquid being not less than 100 c.c. Finally it is concentrated to 60 c.c., when decolorization usually occurs, and a part of the urea is converted into ammonia—but this is of no consequence; if the liquid is not quite clear and colourless, a minute amount of oxalic acid is added. The product is transferred to a vessel which can be cooled, where it is treated with 33 per cent. sodium hydroxide solution, 1 c.c. at a time, until it is alkaline to litmus, and a flocculent precipitate begins to appear, taking care that the temperature does not exceed 15° or 20° C. In a suitable nitrometer, such as that which Jolles has described, this liquid is decomposed with 50 c.c. of a solution containing 80 grammes of sodium hydroxide and 25 grammes of bromine per litre, the evolved nitrogen being corrected and converted into weight by means of the usual tables.

Makowka quotes a series of six normal and pathological urines analysed by the Ludwig-Salkowski and by Jolles' method. The figures show that the latter gives results from 0·8 to 3·1 (mean 2·1) per cent. higher than the former. Inasmuch as the Ludwig-Salkowski process when tested on pure uric acid yields 2 per cent. too little, it follows that Jolles' method is accurate. F. H. L.

A Gold Reagent for Colloidal Substances. R. Zsigmondy. (*Zeit. anal. Chem.*, 1901, xl., 697-719.)—The author's reagent is prepared by heating 120 c.c. of water (distilled through a silver condenser) to the boiling-point, and adding 2·5 c.c. of a solution of gold hydrochloride (6 grammes $AuCl_3.HCl, 3H_2O$ per litre) and 3 to 3·5 c.c. of a solution of pure potassium carbonate (0·18 N.). Immediately after the liquid boils 3 to 5 c.c. of a dilute solution of formaldehyde (0·3 c.c. of formalin in 100 c.c.) are added with vigorous stirring, with the result that a deep red permanent colour is produced.

On mixing 5 to 10 c.c. of this solution with a few drops of a solution of a colloid and adding 0·5 to 1 c.c. of a 10 per cent. solution of sodium chloride, a difference is observed with different classes of colloidal substances.

The author classifies them in the following groups according to their behaviour with the reagent:

1. *Active Colloids.*—Glue, gelatin, casein, isinglass, etc. From 0·005 to 0·1 milligramme of these is sufficient to prevent the change of colour of 10 c.c. of a 0·0056 per cent. gold solution to violet or blue after the addition of 1 c.c. of sodium chloride solution.

2. Gum-arabic, egg albumin, gum tragacanth, carrageen, etc. These are less active, from 0·1 to 10 milligrammes being required to prevent the change of colour.

3. Dextrin, potato starch, etc., of which from 10 to 500 milligrammes are required.

4. *Inactive Colloids.*—To this group belong silicic acid, soluble glass, and other substances, the addition of which does not interfere with the change of colour. On long keeping, however, the restrictive power of most colloidal solutions shows a marked decrease.

The author applies the term *gold value* to the number of milligrammes of a colloid necessary to prevent the change of colour of 10 c.c. of the gold reagent immediately or soon after the addition of 1 c.c. of sodium chloride solution.

He has obtained the following gold values for different colloids: Freshly prepared glue and gelatin solutions, 0·005 to 0·01; gum arabic, 0·1 to 2 or 4; dextrin, about 20; and colloids of the fourth class, nil.

It is pointed out that well-prepared gold solutions undergo changes when kept for a long time, and that under such circumstances they should be standardized on gum arabic or dextrin, the gold values of which are known.

The effect of various factors on the gold value is also discussed. C. A. M.

INORGANIC ANALYSIS.

Determination of Silver in the Residues from the Distillation of Zinc. K. Sander. (*Zeits. f. angew. Chem.*, xv., 32.)—The residues contain a considerable quantity of lead, 20 or 30 per cent. of carbon, and all the silver that was in the ore. In order to remove the carbon rapidly the author oxidizes with a mixture of 80 parts potassium nitrate and 20 parts sodium peroxide. He mixes 20 grammes of the coarsely-powdered residue with 50 grammes of the above mixture, and introduces it in small quantities of 3 or 4 grammes at a time into a red-hot iron crucible. As soon as the reaction is not very violent the crucible is placed in the fire, the necessary quantity of flux is added, and 10 grammes of litharge. The button of lead obtained is cupelled in the usual way. A. M.

The Separation of Copper, Lead, Antimony, and Tin in Alloys. A. Rössing. (*Zeit. anal. Chem.*, 1902, xli., 1-11.)—*Solubility of Copper Sulphide in Alkali Sulphides.*—The author shows in a series of experiments that copper sulphide is soluble in yellow ammonium sulphide and in sodium polysulphide, the respective compounds $(NH_4)_2Cu_2S_7$ and $Na_2Cu_2S_7$ being formed. The loss in the analysis of alloys is greater when the influence of the air is excluded, and in some of the experiments amounted to as much as 5·71 per cent. of the total amount of copper.

On the other hand, accurate results were obtained by using *colourless* sodium sulphide, and the author therefore considers it essential to use that compound in the separation of metals of the hydrogen sulphide group when copper is present.

Thus, since only the alkali monosulphide is applicable for the separation of copper from tin, antimony, and arsenic, care must be taken that the tin has been sufficiently oxidized or chlorinated.

Separation of Lead and Copper from Tin and Antimony.—The method of converting the tin and antimony into insoluble oxides by treatment with nitric acid, whilst converting the lead and copper into soluble nitrates, has not yielded good results in the author's hands. In every instance he found more or less copper and lead in the insoluble residue.

The following method is recommended: Two grammes of the coarsely-divided alloy are dissolved in the smallest possible amount of *aqua regia*, and the solution heated with a little potassium chlorate as a precaution. It is then diluted with water, a little tartaric acid being added, rendered slightly alkaline with sodium hydroxide, and treated with the smallest possible excess of colourless sodium

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sulphide. The liquid is heated for a short time, allowed to stand, and filtered. The residue is washed with water containing a little sodium sulphide, and dissolved in hot dilute nitric acid, and the lead and copper in the solution separated in the usual way.

Separation of Tin from Antimony.—Von Carnot's method, which is based upon the precipitation of the antimony as oxysulphide by sodium thiosulphate,

$$2SbCl_3 + 3Na_2S_2O_3 = Sb_2OS_2 + 6NaCl + 4SO_2,$$

has given inaccurate results in the author's experiments, as was also the case with the modification proposed by Andrews (*Zeit. anal. Chem.*, xxxviii., 796).

Clarke's method, however (*Zeit. anal. Chem.*, ix., 487), which consists in precipitating the antimony from the boiling solution by means of hydrogen sulphide in the presence of much oxalic acid and absence of much free mineral acid, yielded accurate results.

In determining the antimony thus separated the author proceeds as follows: The precipitate is washed with hot water, and then successively with alcohol, a mixture of alcohol and carbon bisulphide (to remove free sulphur), alcohol, and finally ether, and is dried at a low temperature.

It is next detached from the paper, the small residual portion being dissolved off with hot ammonium sulphide, and ignited in a weighed porcelain crucible. The main portion is treated with concentrated nitric acid, which is evaporated on the water-bath, the oxidation being repeated until unoxidized sulphur can no longer be observed. The temperature is then raised, with the precautions noted by Brunck (ANALYST, xx., 190), and the crucible finally heated over the blowpipe until constant in weight. C. A. M.

Valuation of Commercial Aluminium Sulphate. C. R. Gyzander. (*Chem. News*, 1901, lxxxiv., 296 and 306.)—The sample is brought into solution, mixed with a small known quantity of standard sulphuric acid equivalent in strength to the alkali used, and titrated with sodium hydroxide (say of 11·65 grammes of NaOH per litre, which gives direct readings in percentages of Al_2O_3 when 0·5 gramme of sulphate is employed) in presence of methyl orange and phenolphthalein, till the colour changes from pink to orange, not yellow. According to the amount of alkali used, allowing for the acid purposely added, the sample is either basic, normal, or acid. Titration is then continued at 30° C. till a permanent pink is produced, and this gives the total alumina. The object of the methyl orange is to mask the pink tint of the phenolphthalein-alumina lake, which is otherwise troublesome.

Minute amounts of free acid, which cannot be determined by titration, can be estimated by observing the time taken for a solution of the aluminium sulphate to decolorize a "solution" (1 gramme per litre) of a standard sample of artificial ultramarine in comparison with that required by a pure sulphate, or by a specimen thereof to which known quantities of acid have been introduced. F. H. L.

Presence of Arsenic in Aqueous Ammonia. O. Gottheil. (*Pharm. Zeit.*, 1901, xlvi., 992; through *Chem. Zeit. Rep.*, 1901, 365.)—The author has found 0·057 per cent. of As_4O_6 in an ordinary commercial specimen of 25 per cent. ammonia.

F. H. L.

Determinations of the Strength of Aqueous Solutions of Hydrofluoric Acid. F. Winteler. (*Zeits. f. angew. Chem.*, xv., 33.)—All glass beakers, pipettes, rods, or other glass articles which may come in contact with the hydrofluoric acid must be well covered with paraffin wax. The acid should be titrated with standard caustic alkali, using phenolphthalein as indicator (see ANALYST, 1898, 165). The caustic alkali must, of course, be free from carbonate. Using these precautions the author has drawn up the following table of specific gravities of chemically-pure hydrofluoric acid. The figures differ considerably from those of Eckelt (ANALYST, 1898, 193).

SPECIFIC GRAVITIES AT 20° C.

Per cent. HF.	Specific Gravity.	Degrees Baumé.	Per cent. HF.	Specific Gravity.	Degrees Baumé.
1	1·003	0·5	26	1·092	12·0
2	1·007	1·0	27	1·095	12·4
3	1·011	1·5	28	1·098	12·7
4	1·014	2·0	29	1·101	13·1
5	1·018	2·5	30	1·104	13·4
6	1·023	3·0	31	1·106	13·7
7	1·027	3·5	32	1·109	14·1
8	1·030	4·0	33	1·112	14·4
9	1·035	4·5	34	1·114	14·7
10	1·038	5·0	35	1·117	15·0
11	1·041	5·5	36	1·120	15·4
12	1·045	6·0	37	1·122	15·7
13	1·049	6·5	38	1·125	16·0
14	1·052	7·0	39	1·127	16·3
15	1·055	7·5	40	1·130	16·5
16	1·059	8·0	41	1·133	16·8
17	1·062	8·4	42	1·136	17·2
18	1·066	8·8	43	1·138	17·5
19	1·069	9·3	44	1·141	17·8
20	1·072	9·7	45	1·143	18·1
21	1·076	10·1	46	1·146	18·4
22	1·079	10·5	47	1·149	18·7
23	1·082	10·9	48	1·152	19·0
24	1·086	11·3	49	1·154	19·3
25	1·089	11·7	50	1·157	19·5

A. M.

Colorimetric Estimation of Nitric Acid. H. Noll. (*Zeits. f. angew. Chem.*, xiv., 1317.)—After reviewing various methods which have been proposed for estimating nitric acid in water by means of brucine (*cf.* ANALYST, 1881, 56; 1901, 306) the author gives his own: To 10 c.c. of the water add 0·05 gramme of brucine dissolved in 20 c.c. of sulphuric acid (specific gravity 1·840), stir for a quarter of a minute, then pour into a Hehner-Nessler tube which already contains 70 c.c. of water. For comparison a solution is made containing 0·1871 gramme of potassium nitrate per litre. Of this, 10 c.c. = 1 milligramme of nitric acid (N_2O_5). For the determination, 5 c.c. or less are taken, diluted to 10 c.c., and treated exactly as the water under examination, the colours being finally adjusted by running out some of the liquid which is the more strongly coloured. If the water under examination contain more than 50 milligrammes of nitric acid per litre it must be diluted. The results agree fairly well with those obtained by the methods of Marx, Trommsdorf, Schulze-Tiemann, and Henriet. A. M.

A Gas-Volumetric Method of Determining Free and Combined Sulphuric Acid, and Free and Combined Carbonic Acid. E. Riegler. (*Zeit. anal. Chem.*, 1902, xli., 17-25.)

Sulphates and Sulphuric Acid.—A hydrochloric acid solution of the sulphate is precipitated with a known excess of barium chloride solution (30·5 grammes per litre; 1 c.c. = 0·010 gramme SO_3), and the excess of barium chloride precipitated as iodate, which on treatment with hydrazine sulphate yields elementary nitrogen as a product of the reaction. Thus:

1. $BaCl_2 + 2HIO_3 = Ba(IO_3)_2 + 2HCl.$
2. $Ba(IO_3)_2 + 3N_2H_4H_2SO_4 = BaSO_4 + 2H_2SO_4 + 2HI + 6H_2O + 6N.$

The details of the method are as follows: From 30 to 40 c.c. of the solution (which should only contain a very small amount of sulphate) are acidified with 5 drops of concentrated hydrochloric acid, and the sulphate precipitated with 5 c.c. of the standard barium chloride solution, further additions of 5 c.c. each being made until the precipitation is complete. This is shown by testing a drop of the liquid with potassium chromate solution; and it is essential that the excess of chloride shall not exceed 5 c.c., in order to avoid the evolution of too large a volume of nitrogen.

The liquid is boiled, shaken, allowed to cool, shaken with 5 c.c. of a 6 per cent. solution of iodic acid, and left for fifteen minutes.

The precipitate of barium iodate and sulphate is now collected on a filter, and washed with water until the filtrate and washings amount to 100 c.c., after which the filter-paper is rolled up and placed in the inner vessel of a Knop-Wagner nitrometer. The outer vessel receives 40 c.c. of a 2 per cent. solution of hydrazine sulphate, and the nitrogen evolved is collected and reduced to standard pressure and temperature in the usual manner (*cf.* ANALYST, xxvi., 215, 239, and xxvii., 72).

One milligramme of nitrogen corresponds to 0·9504 milligramme of SO_3, or 1 c.c. of nitrogen at 0° C., and 760 millimetres pressure to 1·192 milligrammes of SO_3. A correction, experimentally found to be 0·0035 gramme, is made for the solubility of the barium iodate in the 100 c.c. of washings.

Free and Combined Carbonic Acid.—The method is based upon the conversion of barium carbonate into chloride, the formation of iodate from the chloride, and the liberation of nitrogen by the action of hydrazine sulphate on the iodate.

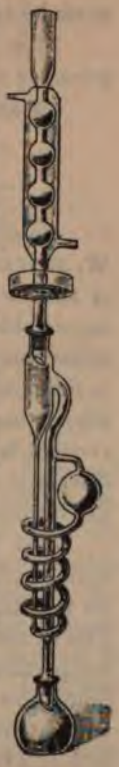
The solution of the carbonate, which must not contain more than 0·025 gramme of carbonic acid, is shaken with a few c.c. of a solution of barium hydroxide, and the resulting precipitate of barium carbonate collected, washed, and treated with about 30 c.c. of water and 15 drops of concentrated hydrochloric acid. After the flask has been well shaken, 5 c.c. of a 6 per cent. solution of iodic acid are introduced, the flask again shaken for fifteen seconds, and then allowed to stand for fifteen minutes.

The precipitated barium iodate is collected, washed, and treated with hydrazine sulphate as described above.

Each milligramme of nitrogen corresponds to 0·5223 milligramme of carbon dioxide, or 1 c.c. of nitrogen at 0° C., and 760 millimetres' pressure is equivalent to 0·6552 milligramme CO_2. The correction for the solubility of the barium iodate in the 100 c.c. of filtrate and washings is 2·1 milligrammes of carbon dioxide.

The experimental results quoted are in close agreement with the theoretical amounts. C. A. M.

The Direct Gravimetric Determination of Boric Acid. A. Partheil and J. A. Rose. (*Berichte*, 1901, xxxiv., 3611, 3612.)—This method is based upon the solubility of boric acid in ether saturated with water, and on its non volatility when the ethereal solution is evaporated *in vacuo* over sulphuric acid.



The authors recommend the apparatus shown in the figure for the determination. The boric acid solution, which has been acidified with hydrochloric acid, is introduced into the spiral tube, but should not reach as far as the bulb. Freshly-rectified ether is next added in sufficient quantity to nearly fill the bulb, whilst 20 c.c. of ether are placed in the weighed flask below the extractor. The extraction is continued on a water-bath for about eighteen hours, and then for a further two hours with a second weighed flask, so as to insure that all boric acid has been removed.

The flask is placed in a vacuum desiccator over sulphuric acid, and the residue dried until constant in weight, and weighed as H_3BO_3. In this way a solution of boric acid containing 0·31015 gramme yielded 0·3105 and 0·31025 gramme, whilst a solution of borax gave 0·12425 gramme as against the theoretical amount 0·12408 gramme.

The authors state that this method is also applicable to the determination of boric acid in minerals and in food-products.

The absence of sulphuric acid, nitric acid, phosphoric acid, iron in large quantity, zinc chloride, or arsenious acid, must be insuréd, since these substances also dissolve in the ether.

The purity of the residue obtained can be tested by repeatedly evaporating it with pure methyl alcohol, and deducting the weight of any non-volatile substance. C. A. M.

The Determination of Small Quantities of Hydrogen Sulphide in Natural Waters. L. W. Winkler. (*Zeit. anal. Chem.*, 1901, xl., 772-774.)—If the water is first treated with a solution of sodium potassium tartrate to prevent the separation of magnesium or calcium carbonate, and then mixed with an alkaline solution of lead acetate, a brownish tint is produced, which can be compared with that given by a standard solution of sulphide under the same conditions.

In making a determination, 100 c.c. of the water are shaken with 5 c.c. of a reagent consisting of 25 grammes of sodium potassium tartrate, 5 grammes of sodium hydroxide, and 1 gramme of lead acetate in 100 c.c. of water.

One hundred c.c. of distilled water and 5 c.c. of the reagent are placed in a similar flask, and a dilute standard solution of ammonium thioarsenite run in from a burette until the colour of the liquid in the first flask is matched.

The standard solution is prepared by dissolving 0·0367 gramme of pure arsenic trisulphide in a few drops of ammonium hydroxide, and diluting the solution to 100 c.c. One c.c. of this solution corresponds to 0·1 c.c. of hydrogen sulphide at 0° C. and 760 millimetres' pressure.

If the amount of hydrogen sulphide in the water is less than 0·2 c.c. per litre, the coloration is too faint, and a larger quantity (500 to 1,000 c.c.) of the sample must be taken.

This method has the advantage that the results are not influenced by the presence of thiosulphates, which are usually present in sulphuretted mineral waters.

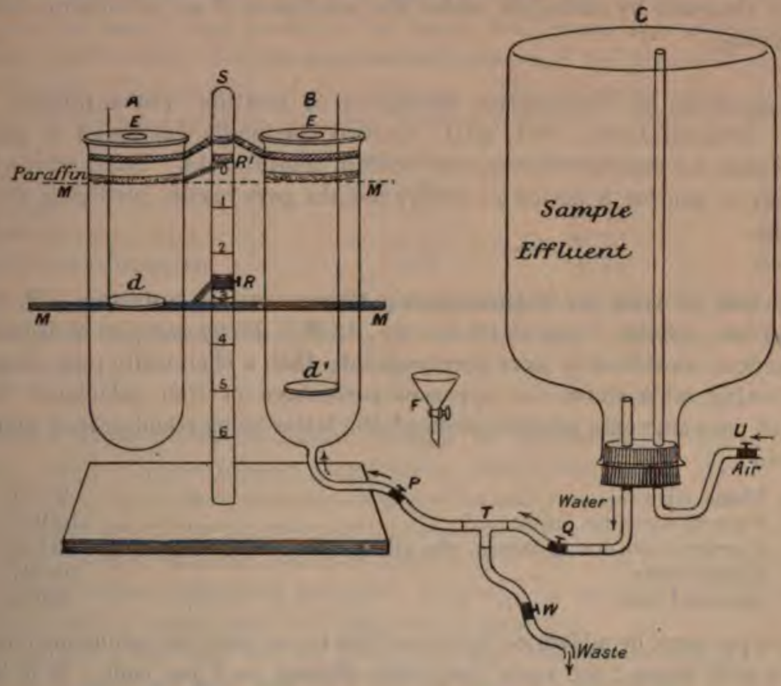
Its accuracy is shown by a series of test experiments. C. A. M.

Colorimetric Method for determining Oxygen dissolved in Water. W. Ramsay and I. Homfray. (*Journ. Soc. Chem. Ind.*, xx., 1071.)—Use is made of the fact that dissolved oxygen oxidizes ammoniacal cuprous chloride solution to cupric chloride, the resulting blue colour being a measure of the oxygen present. The apparatus consists of two glass comparison tubes (A, B), 12 inches long and 2 inches in diameter; they are closed with movable caps (E), through which pass brass wires supporting the opal glass discs (d, d^1). The tubes are supported on a stand with a vertical brass stem (S), which is graduated in inches and tenths. The levels of the discs and of the liquids in the tubes are read off against the scale with the help of the horizontal levelling-rod (M, M), which slides along S.

Just before the experiment the bottle of effluent (C) is unstoppered and fitted with a rubber cork and tubes as shown. Some paraffin oil is poured into B, and the air is entirely displaced from the tubes by manipulating the screw-clips (U, Q, W, P). The effluent is allowed to run into B. Into A is poured distilled water of known temperature saturated with air. This also is covered with a layer of paraffin. Some powdered cuprous chloride is placed in the small tap-funnel (F), and covered with hydrochloric acid; a dark-brown solution results, some of which is run into A and B, avoiding air bubbles. Equal quantities of aqueous ammonia are then added, and the water is stirred gently by means of the discs. The discs are adjusted until, on looking at them through holes in the caps, they appear equally tinted. From their position a simple calculation gives the percentage of saturation, and hence the number of c.c. of oxygen per litre in the effluent.

The cuprous chloride is made by warming a solution of cupric chloride with scraps of copper, pouring into water, filtering off, and washing with boiling water, alcohol, and ether. It should be quite white, and should be protected from light and damp in a stoppered black test-tube.

If much lime be present in the effluent, to prevent turbidity 2 or 3 c.c. of a hot saturated solution of ammonium chloride should be added to both tubes before the other reagents. If the effluent be coloured yellow a trace of alkaline paranitrophenol may be added to the standard water after the other reagents.



Portable Modification.—The test has been arranged in a portable form, all necessary sample-collecting apparatus, reagents and colour standards, being contained in a small wooden box. With this experiments can be carried out at the riverside in a few minutes. The standards of colour consist of six sealed tubes 6 inches long, containing various strengths of strongly ammoniacal cupric chloride. The experiment is carried out, under paraffin oil, in a similarly shaped tube with ground-glass stopper. Light is reflected up through the tubes by an opal glass plate set at 45°. The standards correspond with 1, 2, 3, 4, 5, 6 c.c. of oxygen per litre. It is easy to estimate within 0·5 c.c.

The results obtained by both modifications agree satisfactorily with those obtained by Thresh's method.

To avoid aeration in collecting samples, the can fitted to the testing-box is open at one end, and has a valve opening inwards at the other. The stream can thus be allowed to flow right through, displacing all air. The presence of nitrites makes the results slightly high (see Rideal and Stewart, ANALYST, 1901, 143). A. M.

The Preparation of Sodium Arsenite Solution for Volumetric Analyses. G. Lunge. (*Zeit. f. angew. Chem.*, xiv., 1293.)—Petriciolli and Reuter in a footnote to a recent paper (*Ibid.*, xiv., 1181) stated that, if sodium arsenite be prepared by boiling arsenious anhydride with sodium bicarbonate, some sodium carbonate is formed which will itself absorb iodine. The author finds, however, that the quantity of iodine absorbed by carbonate under the conditions of an iodometric titration is inappreciable. A. M.

Preservation of Volumetric Solutions of Sodium Thiosulphate. F. H. Alcock. (*Pharm. Journ.*, 1901, 491).—Carbon bisulphide was found to act well as a preservative for standard solutions of sodium thiosulphate. Trials with a solution which was in use for a period of twelve months gave results justifying the recommendation. A. G. L.

The Use of Iron for Standardizing Permanganate Solution. H. Ort and W. Klaproth. (*Zeits. f. angew. Chem.*, xiv., 1233.)—Many samples of iron not very impure reduce considerably more permanganate than a chemically pure iron should. The following table shows the apparent percentage of iron calculated from the amount of permanganate solution reduced, the latter being standardized against pure oxalic acid:

Piano wire	99·41
Ferrum metallic. foliat.	100·31
Ferrum metallic. in lamin. pro analysi	100·55
Flower wire	100·07
Reduced iron	99·98

If 0·4 per cent. be added for the impurities in the iron, the results for most of the irons are still worse; the error may then amount to 1 per cent. It is better to standardize with oxalic acid. A. M.

The Standardization of Uranium Solutions for Phosphate Analysis. J. A. Muller. (*Bull. Soc. Chim.*, 1901, xxv., 1000-1002.)—The uranium solution for the volumetric determination of phosphoric acid is frequently standardized on crystalline sodium phosphate, though the readiness with which this salt loses water is a source of error.

The crystalline salt (PO_4HNaNH_4, $4H_2O$) is much more stable; but even that is less stable than the crystalline dicalcium phosphate—$(PO_4)_2Ca_2H_2$, $4H_2O$—which does not vary in weight when exposed to the air or when kept over phosphoric anhydride.

The author therefore recommends a solution of this salt in nitric acid as a suitable standard.

It is prepared by adding di-sodium phosphate little by little to a cold dilute solution of calcium chloride. The gelatinous precipitate soon becomes crystalline, and is then washed and dried on tiles at about 70° C. Theoretically it contains 41·27 per cent. of P_2O_5, whilst the author's preparations, analysed by different methods, were found to contain from 41·29 to 41·55 per cent. C. A. M.

The Solubility of Phosphatic Manures in some Organic Acids. Walter F. Sutherst. (*Chem. News*, 84, 199.)—In comparing the solvent action of organic acids on manures, the sample was extracted for twenty-four hours with solutions of the acids whose total acidity was the same in each case, and the dissolved phosphoric acid determined in a portion of the filtrates. The following table shows the percentages of the total tricalcic phosphate present which were found in solution:

Manure.	Acetic Acid.	Tartaric Acid.	Citric Acid.
Coprolite	12·01	43·41	20·36
Basic slag	42·22	54·41	67·50
Basic superphosphate	65·29	87·38	99·26
Precipitated phosphate	54·15	96·76	88·28

From his results the author concludes that the most satisfactory results on the whole are given by tartaric acid, and not by citric acid, the reagent generally employed. A. G. L.

Notes on some Blowpipe Tests. Joseph W. Richards. (*Journ. Am. Chem. Soc.*, xxiii., 213.)—Approximately quantitative determinations of water, volatile sulphur, etc., may be made by heating a weighed quantity of the material in a closed tube. The upper end of the tube containing the sublimate is broken off and weighed; in the case of water its ends are closed by small corks. The tube is then heated until the sublimate has been volatilized, and again weighed. A sample of göthite containing 10·11 per cent. of water gave 10·28 per cent. in this way.

When applying the flame test for phosphoric acid, the sample should be touched whilst hot with sulphuric acid, and then brought as slowly as possible to the outside edge of the Bunsen flame as low down as possible. For the detection of boron the sample should be held about an inch higher.

For the reduction of refractory minerals on charcoal with soda, it is always advantageous to add borax, especially in the case of tin dioxide.

In testing for fluorine by fusion with potassium bisulphate, Brazil wood paper is not reliable. The fusion is best made in a tube of 5 to 8 millimetres diameter, held nearly horizontal, when a silica ring deposits just above the assay. If the tube is broken off just under the ring, and the upper end held vertically under the nose, the odour of hydrofluoric acid can always be noticed.

To obtain sublimates from some arsenates, it is necessary to mix them with charcoal and soda, place this mixture in an open tube, and play on it with the reducing flame. All mercury compounds are also reduced in this way.

In testing for silica by means of the microcosmic bead test, it should be remembered that in the presence of certain oxides the bead is able to dissolve 30 per cent. of its weight of silica, and fairly large quantities of the sample may have to be taken. Some silicates dissolve as such in the bead, and in this case long blowing (five minutes) is necessary to decompose them and produce a skeleton of silica.

A. G. L.

Potassium Percarbonate as a Reagent. **F. P. Treadwell.** (*Chem. Zeit.*, 1901, xxv., 1008.)—Potassium percarbonate can now be procured commercially about 80 per cent. pure, and containing only small quantities of chloride and sulphate. As it keeps indefinitely in the dry state, it is a most useful substance from which to prepare hydrogen peroxide—by dissolving it in cold dilute acid—when wanted. The solution obtained by dissolving it in weak sulphuric acid is specially suitable in testing for titanium, vanadium, chromium, cerium, and for the oxidation of ferrous salts. Potassium percarbonate is also applicable in many cases where alkaline hydrogen peroxide is usually employed; it oxidizes sulphuretted hydrogen, soluble sulphides, and the lower sulphur acids to sulphuric acid; it converts manganese, nickel, and cobalt salts to higher oxides. It converts chromium salts into chromic acid, but its action here is not complete, like that of hydrogen peroxide. The percarbonate quickly reduces hypochlorites and hypoiodites to chlorides and iodides. The makers are the Neuhausen Aluminium Co.

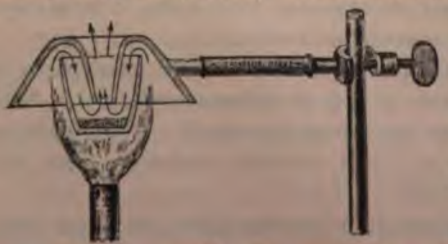
F. H. L.

Use of Oxygen in the Ignition of Precipitates. **H. Zöpfchen.** (*Chem. Zeit.*, 1901, xxv., 1008.)—Whenever a filter bears a precipitate which causes the carbon of the paper to be slow in burning off it may with advantage be placed, while still damp, in a porcelain, or preferably platinum Rose crucible, and ignited in a gentle current of oxygen. The process saves much time, and avoids all danger of any reducing action.

F. H. L.

APPARATUS.

A Heat Concentrator for Ignitions and Concentrations. **C. Jung.** (*Chem. Zeit.*, 1901, xxv., 1113.)—The construction and object of this device are sufficiently explained by the diagram. It introduces a current of hot air and combustion products into a crucible, and vastly hastens ignition, etc. By modifying somewhat the shape of the deflector, returning upwards the central part so as to form a short chimney, it can be placed over an evaporating basin, and renders the whole a kind of surface-evaporator as well. The apparatus is made by W. J. Rohrbeck's Nachfolger of Vienna.



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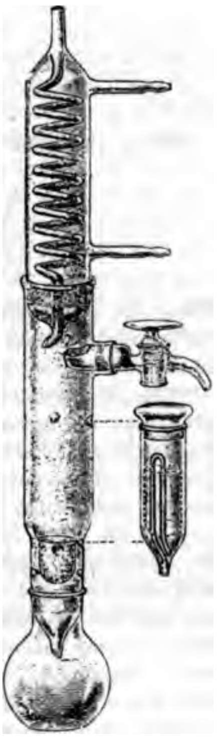
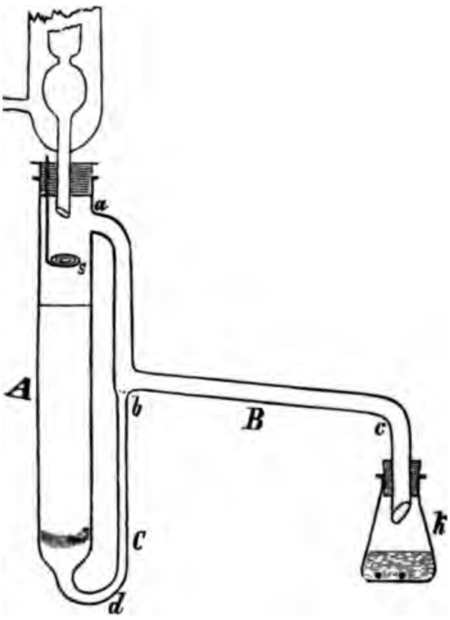


Combined Apparatus for Extraction and Recovery of e Solvent. A. Chatelan. (*Chem. Zeit.*, 1901, xxv., 612.)—s shown by the accompanying sketch, this apparatus is conructed with a ground joint between the extraction vessel and e condenser, the lower opening of the latter being bent to e side. When extraction is finished, the condenser is tated through an angle of 180°, so that the volatile liquid lling from the worm drops into the opening at the inner end the lateral stop-cock. This cock being opened, the solvent n be collected in any suitable receiver. During extraction e cock is closed. The material to be extracted is weighed in e small separate bottle, which is provided with a siphon, d also with a flat stopper to serve as a base when it is on e balance.

F. H. L.



An Apparatus for Extracting Liquids with Chloroform. F. Pregl. (*Zeit. anal. Chem.*, 1901, xl., 785-787.)—The construction of the apparatus devised by the author for the extraction of alkaloids by Kippenberger's method is clearly shown in the accompanying figure. The drops of chloroform falling from the condenser are broken up into smaller drops by the wire spiral *S*, thus facilitating the extraction.

C. A. M.

MILK-BLENDED BUTTER.

KING'S BENCH DIVISION.

(Before the Lord Chief Justice of England, Mr. Justice Darling, and Mr. Justice Channell.)

PEARKS, GUNSTON, AND TEE (LIMITED) *v.* HOUGHTON.

From the "Times" of February 27, 1902.

This was a case stated by the Justices of Richmond, Surrey, raising a question as to the sufficiency of certain notices in reference to milk-blended butter within the meaning of Section 8 of the Sale of Food and Drugs Act, 1875.

The following facts appeared from the case stated: An information was preferred by Houghton, the respondent, against Pearks, Gunston, and Tee (Limited), the appellants, under Section 6 of the Sale of Food and Drugs Act, 1875, charging them with unlawfully selling to the prejudice of the purchaser half a pound of shilling butter which was not of the nature, substance, and quality demanded. having had water added to it to the extent of 7·8 per cent. beyond the usual limit of 16 per cent. natural to butter. The appellants were convicted and fined £20 and costs. The appellants were grocers and provision merchants at Richmond and elsewhere, and the respondent was an inspector under the Act. The respondent sent a boy into the appellants' shop to buy half a pound of shilling butter for the purpose of analysis. The purchase was made and the respondent entered the shop, and the butter was handed to him. The Public Analyst certified that the butter contained 23·8 per cent. of water. The butter was handed out by the shop assistant, wrapped in two pieces of paper. On the inside wrapper there were printed in large type the words "Pearks' Butter," and underneath, in much smaller type, a statement that the butter was blended with pure English full-cream milk by new and improved machinery, whereby it retained about 20 to 24 per cent. of moisture. The respondent's attention was not called by the shop assistant to what was on the inside wrapper, and the respondent, though he saw it, did not read it. It was proved by the appellants that it was a common practice in the trade to blend different kinds of butter, and that the process of blending adopted by the appellants was to put the butter into a churn with full-cream milk, and this was re-churned. Any excess of water in the butter in question was derived solely from the milk so added during this process of blending. No water was separately added to the butter. It was stated in evidence on behalf of the appellants that the object of blending the butter with milk is to give it uniform colour and flavour and freshness, but there was no evidence that the milk was added because it was required for the preparation of the butter as an article of commerce fit for carriage or consumption. The price of full-cream milk is ¾d. a pound. In a frame on the wall behind the butter-counter in the appellants' shop was a printed notice to the same effect as that on the wrapper. The butter-counter faces the front of the shop, and the notice was visible to anyone going into the shop; but the respondent did not observe it, and his attention was not called to it. It was contended for the appellants that the inside wrapper was a label within Section 8 of the Sale of Food and Drugs Act, 1875, that the printed notice in the shop was sufficient notice to the respondent apart from the label, and that there was no evidence of fraud. It was further contended that, as the wrapper and printed notice accurately stated the nature and composition of the article sold, there was no evidence of a sale to the prejudice of the purchaser within Section 6 of the Act. It was contended for the respondent that the sale was complete before he entered the shop, and that notice was then too late, and that, as the butter was delivered in a piece of plain, opaque paper, which prevented the printing on the inside wrapper being visible until removed, and as the respondent did not read it, and did not purposely abstain from reading it, and his attention was not called to it, it was not notice within Section 8, and that as he did not observe the notice on the wall, and his attention was not called to it, it was no notice to him at all. The justices were of opinion that the printed matter on the inside wrapper was misleading, and therefore not sufficient notice within Section 8, as it stated the result of the blending to be to "retain" in the butter about 20 to 24 per cent. of moisture, whereas the true result was to "add" 7·8 per cent. of water to the extreme limit of 16 per cent. natural to butter. They also held that for the same reasons the notice on the wall was not sufficient, that the notice on the inside wrapper was not sufficient within Section 8, that, in the circumstances, the notice on the wall was no notice to the respondent, that the notice was too late, and that the excess of water was added fraudulently to increase the bulk and weight, and notice was therefore no protection. The questions for the court were: (1) whether the inside wrapper was a sufficient notice by label within Section 8; (2) whether there was sufficient notice otherwise than by label; and (3) whether there was any evidence of fraud.

Mr. Asquith, K.C., Mr. Avory, K.C., and Mr. H. D. Bonsey were for the appellants; Mr. Alex. Glen was for the respondent.

Mr. Asquith, for the appellants, cited "Sandys *v.* Small" (3 Q.B.D., 449), "Jones *v.* Jones" (58 J.P., 653), "Spiers and Pond *v.* Bennett" (1896, 2 Q.B., 65), and "Pope *v.* Tearle" (L.R. 9, C.P., 499). He contended that the notices were sufficient, and that there was no evidence of fraud.

Mr. Glen, for the respondent, submitted that there was evidence to support the finding of fraud, as the onus in this respect was on the appellants under Section 24 of the Act. He cited "Pearks *v.* Knight" (1901, 2 K.B., 825), "Liddiard *v.* Reece" (44 J.P., 233), and "Horder *v.* Meddings" (44 J.P., 234).

The Lord Chief Justice, in giving judgment, said that the case was not stated in a satisfactory way; and he did not express any opinion that, if the court was satisfied that this article had been sold as butter and nothing else, there would not be evidence of an offence under Section 6. That was practically decided in "Pearks *v.* Knight," and his Lordship would have come to the same conclusion. But that was not the way in which they had to regard this case. The butter was asked for as half a pound of 1s. butter. If it should turn out that there were various classes of butter in the establishment, and that this was merely butter at a certain price, a different effect had to be given to the notice put up in the shop. He understood the facts to be that the appellants were selling one sort of butter only, by which he meant blended butter. That was the conclusion he drew from the facts, and he did not intend anything he said to apply to a man who sold different kinds of butter and put up this notice. Speaking for himself only, he was not satisfied that if the case had been made out of an offence under Section 6 the inner label would have protected the appellants, but he did not think that was the essential point. If they were dealing with Section 8 only they would have to consider to a greater extent the words of the section "intended fraudulently to increase its bulk," and say whether the person was supplied with a notice at the time. For himself, he had a doubt whether on the delivery of such an article a notice on the inside wrapper was sufficient. "Jones *v.* Jones" was not a sufficient authority for the appellants on that point. He did not think that any one buying a pound of butter and having it delivered to him would be held to have general notice that there was another label inside. If a man had asked for butter and got the article now in question, and the only defence was the label, he was not prepared at present to say that the appellants' contention was right. He understood the magistrates to find that the ordinary purchaser would see the notice on the wall, and he did not think this was qualified by the statement that the respondent did not see it. They were bound by the case of "Sandys *v.* Small" and "Spiers and Pond *v.* Bennett," which decided that if the purchaser was told, not necessarily orally, at the time of the purchase that the article sold was a blended article, it could not then be said that it was sold

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to the prejudice of the purchaser. No question of Section 24 and the onus of proof arose here. The prosecution had to prove that there was a sale to the prejudice of the purchaser. It was good law and good sense that, when a man was told that what he was buying was butter blended with something else, he was not entitled to say that he was prejudiced because he thought he was buying butter not blended. To a person who understood the chemistry of butter it would be wrong to say that it "retained" 20 to 23 per cent. of water, unless there was a discussion as to what it was mixed with. To the ordinary purchaser the notice contained a statement that the butter was mixed with milk and had in it 23 per cent. of moisture. He did not think that any supposed misleading statement in that notice would do away with the broad statement that the butter sold in that establishment was in accordance with the notice. He thought there was no evidence of an offence on the ground of prejudice to the purchaser. The magistrates had found that the excess was added fraudulently to increase the bulk and weight. But, if there was no offence under Section 6, the question of supposed fraudulent addition did not arise. He could see no evidence of fraud, but he would hesitate to overrule that class of finding if it was essential to listen to it in order to arrive at justice in a case of the kind.

The other learned JUDGES concurred, and the appeal was accordingly allowed and the conviction quashed, with costs.

REVIEW.

LABORATORY COMPANION TO FATS AND OILS INDUSTRIES. By Dr. J. LEWKOWITSCH, M.A., F.I.C. London: Macmillan and Co. Price 6s. net.

The book, which consists principally of tables, is essentially a companion to the author's "Chemical Analysis of Oils, Fats, and Waxes." Part I. contains, in addition to a number of tables, some pages of matter explanatory of the author's system of fats and oils, which he considers to have been considerably strengthened since the appearance of his "Chemical Analysis." Part II. consists of a number of tables giving the constants of fats, oils, waxes, and the commercial products derived from them, and this will no doubt be found the most useful portion of the book. Part III. contains tables to which frequent reference has to be made in the course of the analysis of fats, oils, etc. The work will be extremely serviceable to those engaged in this special branch of analysis. W. J. S.

BRITISH COLUMBIA INSTITUTE OF ASSAYERS.

AT a provisional meeting held at Nelson, British Columbia, on November 8, 1901, it was unanimously resolved that an Association of Assayers should be formed with the above title, having the following objects in view: The study and promotion of assaying and metallurgical chemistry, and the advancement of the profession generally. No candidate is to be admitted as a member of the Institute unless he is certificated under the Bureau of Mines Amendment Act of 1899. The following officers have been elected for the session 1901-02: President—H. Carmichael, Government Assayer, Victoria. Vice-President—A. McKillop, Nelson. Secretary-Treasurer—J. Cuthbert Welch, Trail. Ordinary Members of Council—A. A. Cole, Rossland; H. Harris, Nelson; Thos. Kiddie, Van Anda; Wm. F. Robertson, Prov. Mineralogist, Victoria.

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VOLUME XXVII.—No. 313.
APRIL, 1902.

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NOTE ON REICHARDS "SILVER" METHOD FOR THE DETERMINATION OF MORPHINE IN OPIUM.

By Philip Schidrowitz, Ph.D.

(*Read at the Meeting, January* 22, 1902.)

Reichard (*Chemiker Zeitung*, xxv., 816, 817) has devised a process for the estimation of morphine in opium, which consists essentially in the determination by weighing of the metallic silver obtained by the reducing action of the alkaloid on an ammoniacal silver chloride solution. According to Reichard, morphine is the only one of the opium alkaloids which—at any rate, under the conditions of his process—exercises a reducing action on a silver solution. Reichard further states that, if other reducing substances be present in the opium, their influence can be eliminated by precipitating the "alkaloids (morphia)" (*sic*) with ammonia, filtering, treating the filtrate with ammoniacal silver chloride, weighing the reduced silver, and subtracting the weight so obtained from the gross amount. Reichard, however, states that after precipitation with ammonia he was never able to observe any reduction. Neither of these assertions bears the stamp of probability, but, owing to lack of time, I was not able to enter into this aspect of the question, but confined myself to a practical trial of the method. I found, in the first place, that if Reichard's instructions were literally followed, it was, owing to the formation of colloidal silver, practically impossible to satisfactorily wash the precipitated silver; but this difficulty was overcome by employing a 20 per cent. solution of chloride of ammonium (with the addition of a little ammonia) as wash liquor. Contrary to Reichard's experience, I found that, after precipitation with ammonia and filtration, the filtrate exercised a strong reducing action on the reagent prescribed, and here also it was necessary to make use of a liberal amount of ammonium chloride (added to the liquor before filtration) in order efficiently to filter and wash the precipitate. The sample worked on was a good average Persian opium, which had been repeatedly analysed by several of the more reliable methods, and which gave (as an average of three methods) 10·98 *per cent.* morphine. At first I worked on 5 grammes of opium, but found that the quantity of silver precipitated was too large to be conveniently handled, and the results given below were all obtained by operating on 2 grammes of the drug. The ammoniacal silver solution used contained roughly 5 per cent. AgCl, and 5 c.c. should, therefore, have (according to Reichard) been amply sufficient for a complete precipitation. The results obtained by the direct method were as follows (Table I.):

Table I.

(*Two Grammes of Opium.*)

No. of Experiment.	No. of c.c. Silver Solution.	Weight of Precipitated Silver.	= Morphine per cent.
1	5	0·15049	10·54
2	7	0·21189	14·85
3	8	0·25369	17·77
4	16	0·39609	27·75
5	30	0·29929	20·98

TABLE II.

(*Results* AFTER *Precipitation with Ammonia.*)

No. of Experiment.	No. of c.c. Silver Solution.	Weight of Precipitated Silver.
6	5	0·1459
7	10	0·2591
8	20	0·2189

Up to a certain point, therefore, it appears that the quantity of silver reduced is not dependent on the morphine contents, but rather on the quantity of silver solution employed. It is curious that in both series a very large excess of silver solution gives *lower* results than those obtained by a more moderate addition. It is obvious that in its present form, at any rate, the method is quite useless.

SPURIOUS CREAM OF TARTAR.

By JOHN WHITE, F.I.C.

(*Read at the Meeting, January* 22, 1902.)

SOME few months ago a sample purchased as cream of tartar, and consequently labelled "Cream of Tartar," was submitted to me for analysis under the Sale of Food and Drugs Act.

The British Pharmacopœia, 1898, states that each gramme of dry cream of tartar should require for neutralization at least 5·2 c.c. of normal NaHO, which corresponds to 97·76 per cent. $KHC_4H_4O_6$. Two grammes of the sample I am describing required 9·6 c.c. normal NaHO = 4·8 for 1 gramme, corresponding, apparently, to 90·24 per cent. As shown in a paper read by A. H. Allen (ANALYST, xxi., 174), the alkalinity of the soluble ash of pure cream of tartar should be exactly equivalent to the acidity of the original substance. This sample, however, after being ignited and the ash boiled out with water, required only 1·1 c.c. normal H_2SO_4 to neutralize the solution, a result which conclusively proved it to be a fictitious article.

The sample was not completely soluble in boiling water, the solution being turbid, and a small residue remaining; this was cleared up by a little HCl. Upon testing qualitatively, reactions were obtained for lime, sulphuric and phosphoric acids, and, most surprisingly, for starch. The starch was identified by the microscope as that of rice, and finally, after comparison with known standards, I arrived at the conclusion that there was approximately 5 per cent. present.

A complete analysis of the substance was made, when the following results were obtained:

Moisture	4·10
Silica	0·30
Potassium oxide (K_2O)	15·50
Lime (CaO)	9·10
Tartaric acid ($H_2C_4H_4O_6$)	47·07
Phosphoric anhydride (P_2O_5)	6·97
Sulphuric anhydride (SO_3)	12·70
Rice starch	5·00
	100·74

The tartaric acid was estimated by a modification of Goldenberg's method, which is abstracted in the ANALYST (xxi., 333). I made some slight alterations in details, and, as the method proved to be both quick and of sufficiently practical accuracy, I state it as adopted: Two grammes of the substance are dissolved in 20 c.c. of boiling water, together with 5 c.c. of dilute HCl. The solution is made up to 100 c.c., of which 50 c.c. are made faintly alkaline by the addition of powdered K_2CO_3. Boil for two minutes, cool and make up to 100 c.c. Of this liquid, after filtration if necessary, 50 c.c. (= 0·5 gramme original substance) are transferred to a 500 c.c. flask, 10 c.c. glacial acetic acid added, and then 250 c.c. of a mixture of equal parts of alcohol 90 per cent. and ether 0·730, and the flask closed with a well-fitting cork. The whole is then well shaken, hence the use of so large a flask, and allowed to stand for about three hours, in which time the potassium hydrogen tartrate formed is completely precipitated (I believe one hour would be sufficient for the purpose). Filter, wash the precipitate with the alcohol and ether mixture until neutral to litmus, remove it from the filter, dissolve in boiling water, washing off also the adhering traces from the filter-paper, and finally titrate with $\frac{N}{10}$ NaHO. The following figures show the process to give fairly satisfactory results:

	Direct Titration.	Modified Goldenberg's Method.
Cream of tartar	98·9	99·4
Tartaric acid	97·8	96·4

In order to ascertain whether this method would be affected by the presence of potassium hydrogen sulphate, a mixture of equal parts of cream of tartar (98 per cent.) and potassium hydrogen sulphate was examined. The result obtained was a little low, viz., 48 per cent. cream of tartar.

A consideration of the whole of the analytical results yielded by the sample points to the probability that the material was originally made by the adulteration of cream of tartar with bone-ash superphosphate and starch. The amount of K_2O present is equal to 62 per cent. of cream of tartar. Assuming this to be of 97 per cent. purity, the proportions in which the ingredients were mixed is probably as follows:

Cream of tartar = 64
Superphosphate of lime = 31
Rice starch = 5

100

There is a considerable amount of ingenuity shown in the manufacture of such an article as this, as, although actually containing only 64 per cent. of cream of tartar, when tested by direct titration a result is obtained corresponding to over 90 per cent. This is of course due to the acidity of the added superphosphate.

It is much to be regretted that the compilers of the last edition of the Pharmacopœia, when adopting the method of direct titration of cream of tartar with NaHO, as a test of purity, did not also include at least the determination of the alkalinity of the soluble ash. The employment of these tests in conjunction was advocated by A. H. Allen in the paper already referred to, and it is obvious that a much more satisfactory assay of a tartar can be made when both tests are used than when either test is used singly.

Within the last few years I have had occasion to examine a number of samples of cream of tartar, and among them have found a few containing from 3 to 9 per cent. of tartrate of lime. I have also met with cream of tartar substitutes, which have proved to consist entirely of potassium hydrogen sulphate.

In view of the peculiar composition of this sample, and of the fact that at some time or other it must have been a marketable article, I have thought it desirable to lay the results before you.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

Estimation of Fat in Food-stuffs. C. Beger. (*Chem. Zeit.*, 1902, xxvi., 112.)—Nerking and Dormeyer have already shown that an extraction with ether for twelve hours is not sufficient to recover all the fat (or matter really soluble in ether) from various kinds of fodder, and that the yield is larger if the substance is treated with pepsin. In order to study the effect of different methods of extraction, the present author has estimated the proportion of fat in a considerable number of food-stuffs in four different ways: (1) By the usual twelve-hour extraction; (2) by two consecutive extractions of twelve hours each; (3) by two consecutive extractions of twelve hours, the material being powdered and mixed up between the two stages; (4) by peptonizing either one of the above residues or the original sample, and extracting with ether both the portion soluble and that insoluble in water. Taking process 1 as the standard, a table accompanying the paper shows that process 2 does not give an appreciably larger yield of fat, the increase ranging between 0·03 per cent. in the case of palm-kernel cake and 0·28 per cent. in that of linseed, while sesame cake gave an excess of 0·35 per cent. Process 3 gave increases of 0·3 to 0·4 per cent., except in two cases where the yields were practically the same. The peptonizing process led to very different results. If 0·3 per cent. is accepted as the limit of experimental error in a fat determination, many of the samples remained within that limit, others gave increases of 0·35 to 0·51 per cent.; malt showed an additional yield of 0·7 per cent., then came cottonseed cake, flesh meal, poppy cake, and beer dregs with 1·14 per cent. Rice gluten showed the most noticeable difference, with an increase of 5·74 per cent., so that if the yield by process 1 (0·85 per cent.) is called 100, that by the Dormeyer process becomes 675. Similarly, in the case of beer dregs and malt, the additional amount of fat recovered after peptonization was 12 per cent. and 44·8 per cent. respectively.

The usual qualitative tests were applied to these secondary quantities of fat, and showed them to be really fat, especially in the case of gluten. The extra material obtained from cotton meal was a black resin-like substance, and scarcely deserved the name of fat. F. H. L.

Starch as an Adulterant or Drier in Butter. G. E. Patrick and D. Stuart (*Science*, January 24, 1902) presented, at a recent meeting of the Chemical Society of Washington, the results of some analyses of butter samples which show that notable quantities of starch (probably potato starch) are now being used in this food, presumably as a drier. A tinned butter contained about 15 per cent. of glucose and 3 to 5 per cent. of starch. The presence of small amounts of sucrose was indicated, but as the glucose added to butter is in the form of the syrup, which may contain some added sucrose, the latter is probably accidental. The glucose is added as a preservative, and also to improve appearance. H. L.

A New Method for the Determination of Potato Flour in Yeast. A. Hebebrand. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1902, v., 58-61.)—From 0·5 to 1 gramme of the yeast containing potato starch is rubbed down with 20 c.c. of a 7 per cent. solution of sodium hydroxide, and a stream of chlorine is passed through the mixture for one minute at the rate of 4 to 5 bubbles per second; 150 c.c. of distilled water are now added, and the liquid allowed to settle for half an hour. After this time the supernatant liquid, in which the yeast remains suspended, is decanted from the sediment, which is washed three times by decantation with water. The sediment is brought on to a weighed filter, thoroughly washed with water, and then in succession with alcohol, ether, and petroleum spirit. The filter and its contents are dried for an hour at 100° to 105° C. and weighed. The starch is thus obtained as a pure white powder, and, assuming that the added potato flour originally contained 80 per cent. of starch, the method gives accurate results. W. P. S.

The Chemical Analysis of the Apple and some of its Products. C. A. Browne. (*Journ. Amer. Chem. Soc.*, 1901, xxiii., 869-884.)—The composition of the edible or fleshy portion of apples is shown in the following analyses:

	Water.	Reducing Sugars.	Sucrose.	Starch.	Ash.	Acid as Malic.	Marc.
Unripe apples (two analyses)	80·67	6·43	2·84	3·92	0·27	1·14	
Summer apples (six analyses)	85·00	7·10	3·36	1·04 (1 analysis)	0·28	0·68	
Winter apples (twenty-one analyses)	83·16	8·16	4·16	—	0·26	0·59	1·85 (8 analyses)

The starch completely disappears in the ripe fruit. The average amount of cellulose was found to be 0·90 per cent.; of pentosans, 0·50 per cent.; of lignin, 0·40 per cent.; of pectin bodies, 0·40 per cent.; and of proteids, 0·10 per cent.

Apple-juices, ciders, and vinegars gave the following figures on analysis:

	Specific Gravity.	Solids.	Reducing Sugars.	Sucrose.	Free Acid as Malic.	Ash.	Pectin.	Proteids.	Rotation Ventzke, 400-millimetre tube.
Juice from summer apples (five analyses)	1·0502	12·29	6·76	3·23	0·72	0·29	0·12 (4 analyses)	0·03 (4 analyses)	−26·67
Juice from winter apples (four analyses)	1·0569	13·96	8·57	3·40	0·43	0·27	0·12 (1 analysis)	0·02 (1 analysis)	−45·15
"Second pressings"	1·0376	9·14	6·87	1·49	—	0·20	—	—	−31·94
Cider (six analyses)	1·0006	2·34	0·32	—	0·25	0·29	0·04	0·02	− 2·34
Vinegar (four analyses)	1·0184	2·00	0·52	—	0·14	0·44	0·17	0·01	− 2·01

The free acid given above for cider and vinegar represents the malic acid only. The cider further contained 0·61 per cent. of acetic acid, and the vinegar 6·19 per cent. The average amount of alcohol in the ciders was 5·51 per cent. The "second pressings" are obtained by wetting apple pomace with water and re-pressing. This juice is extensively used for jelly-making and vinegar stock.

Other apple products gave results as under:

	Water.	Reducing Sugars.	Sucrose.	Ash.	Free Acid as Malic.	Proteids.	Pectin.	Marc.
Evaporated apples (two analyses)	27·61	32·80	19·02	1·10	4·08	0·87	—	5·53
Apple butter (one analysis)	52·58	37·20	1·14	0·97	2·52	0·25	2·15	1·14
Cider jelly (one analysis)	44·53	49·50	2·18	1·39	3·61	—	1·60	none
Apple pomace (one analysis)	70·76	8·09	2·40	0·49	—	1·25	—	—

W. P. S.

The Bromine Test for Strychnine. J. C. Wharton. (*Journal of Pharmacology*, 1901, viii., 201; through *Chem. Zeit. Rep.*, 1902, 41.)—If the substance is in chloroform solution, it is brought into a small test-tube and placed inside a larger tube charged with boiling water till the solvent has evaporated. The residue, or the original sample, if solid, is then moistened with 2 or 3 drops of 1 : 1 sulphuric acid, put back into its hot-water jacket, and agitated till solution is effected. The mouth of a flask containing bromine is next held over that of the tube, so that the latter may become full of vapour. The tube is well shaken to cause absorption of the bromine, and the excess is driven off by immersion in the hot-water tube. If strychnine is present in considerable quantity, carmine-red vapours will probably appear in a few minutes, increasing in intensity as the bromine evaporates. The colour disappears after a time. If only a little strychnine is present, only a little bromine should be employed, and the halogen itself may be replaced by a solution containing about 1 drop of bromine in 2 or 3 c.c. of chloroform. F. H. L.

The Identification and Properties of α- and β- Eucaine. C. L. Parsons. (*Journ. Amer. Chem. Soc.*, 1901, xxiii., 885-893.)—These two alkaloids have been recently placed on the market for use as local anæsthetics. They closely resemble cocaine, and are often substituted for this drug.

α-Eucaine, or *n*-methylbenzoyltetramethyl-γ-oxypiperidin carbonic acid methyl ester, forms a hydrochloride when treated with hydrochloric acid, melting at about 200° C., but decomposing at the same temperature; α-eucaine melts at 103° C. The hydrochloride is soluble in 10 times its weight of water, the solubility varying with the temperature. It is soluble in its own weight of alcohol. Potassium iodide gives, in even moderately dilute solutions, a white silky precipitate with α-eucaine salts. β-Eucaine and cocaine give no reaction. Ammonia precipitates α- or β-eucaine or cocaine, but α-eucaine is almost insoluble in excess. Potassium dichromate, in strong solution, throws down a fine yellow precipitate when added to solutions of α-eucaine.

β-Eucaine, or benzoylvinyldiacetonalkamine, melts at 91°, and forms a hydrochloride melting at 268° C. At ordinary temperatures the hydrochloride dissolves to the extent of 3 per cent. in water and about 11 per cent. in alcohol. It is almost insoluble in ether. This comparative insolubility is one of its chief characteristics.

Cocaine may be distinguished from either of the above alkaloids or from both by the following reactions. If a small amount of cocaine hydrochloride be rubbed up with dry mercurous chloride, and then moistened with alcohol, it rapidly turns a grayish black. α-Eucaine hydrochloride slowly becomes dark gray, whilst the β-eucaine salt is not affected. Platinic chloride gives a yellow crystalline precipitate with a 1 per cent. solution of cocaine hydrochloride, which is insoluble in hydrochloric acid. α- and β-eucaine hydrochlorides in 1 per cent. solution yield no precipitate. In stronger solutions a precipitate is formed, which, however, dissolves at once in hydrochloric acid. A drop of potassium permanganate solution is at once turned brown by a drop of the eucaine solutions, whilst with cocaine hydrochloride the colour of the permanganate solution is not changed under half an hour; but it also eventually becomes brown. Excess of permanganate should be avoided. Solutions of cocaine hydrochloride in either water or alcohol polarize light strongly to the left. Eucaine solutions do not polarize light. Cocaine when used in the eye always causes mydriasis. β-Eucaine does not dilate the pupil.

The hydrochlorides are easily identified under the polarizing microscope. α-Eucaine hydrochloride crystallizes in little spots, which under polarized light look like highly-coloured rosettes made of very small crystals, the field never showing any constant extinction directions. β-Eucaine hydrochloride crystallizes in feathery or fern-like forms, sometimes blade-like or tabular. The extinction direction is usually slightly oblique to the main axis of the crystal. Rarely the crystals are diamond shape. Cocaine hydrochloride crystallizes in fanlike shapes. Extinction is parallel and perpendicular to the main axis of the crystals. The colours are brilliant and the whole field is characteristic.

In other respects, especially with regard to the usual alkaloid reagents, these three alkaloids behave alike. W. P. S.

The Behaviour of Calcium Hypophosphite in the Animal System. **T. Panzer.** (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1902, v., 11-14.)—The author has made experiments, both on himself and on dogs, regarding the retention of hypophosphites in the body when taken by the mouth. In the case of dogs it was ascertained that these salts were eliminated from the body, principally by the the urine, in twenty-four hours. In the experiments on himself, the elimination appeared to be rather more protracted. As hypophosphites give the same reaction as phosphorous acid when tested by the hydrogen flame-test, in cases of suspected phosphorus-poisoning, it should be ascertained, if possible, whether the deceased had been taking hypophosphites medicinally immediately before death took place. If none had been administered for two or three days before death, and a reaction be obtained, phosphorus-poisoning may then be suspected. W. P. S.

TOXICOLOGICAL ANALYSIS.

The Detection of Morphia in Decomposing Bodies. **T. Panzer.** (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1902, v., 8-10.)—The author found traces of morphia in two corpses, and, for the purpose of ascertaining the stability of morphia in the presence of decomposing matter, kept portions of the bodies for six months (from June to December), morphia being again looked for after this period. The stomachs, portions of the intestines, kidneys, livers, and urine were stored in loosely-covered vessels at the ordinary temperature of the room. Although intense decomposition had taken place, morphia was plainly detected. The following method was employed: The decomposed matter was diluted with water, acidified with tartaric acid, and gently warmed on the water-bath for some hours. After cooling and filtering, the filtrate was evaporated to a syrup, precipitated with alcohol, and again filtered. The alcohol was driven off from the filtrate, the residue dissolved in water, filtered from insoluble matter, and shaken out with ether. The aqueous liquor was then made alkaline with potash and again shaken out with ether. A third extraction with ether was made after acidifying with hydrochloric acid and making slightly alkaline with ammonia. The morphia was now extracted from the liquor by shaking with amyl alcohol. On evaporating the amyl alcohol a brown, sticky residue was obtained. This was dissolved in cold dilute hydrochloric acid and the solution extracted with amyl alcohol, which removed almost the whole of the colouring matter. The acid liquor was rendered alkaline with ammonia and shaken out with acetic ether, which had previously been washed with water. On allowing the acetic ether to spontaneously evaporate, an amorphous residue was obtained, dissolving completely in hydrochloric acid. After standing, this acid solution deposited crystals which gave the usual reactions of morphia. W. P. S.

Destruction of Organic Matter in Toxicological Analysis. G. Meillère. (*Journ. Pharm. Chim.*, 1902, xv., 97-99.)—The following modification of Gautier and Poutet's methods is stated to effect the complete destruction of 250 c.c. of any organic tissue within two hours.

This quantity of the organic matter is finely divided, and mixed with 5 grammes of potassium sulphate and 100 c.c. of a mixture of 1 part of sulphuric acid and 4 parts of nitric acid. The porcelain basin is next cautiously heated until the organic matter has become liquid, after which a further quantity of the acid mixture is introduced into the centre of it by means of a dropping funnel with a stopcock regulated so that 200 c.c. are delivered within an hour. From time to time 1 c.c. of the liquid is evaporated to dryness, and if the residue turns black the addition of acid must be continued.

According to the author, this method is applicable to the detection of lead, arsenic, copper, and zinc in animal or vegetable tissues.

For the determination of total phosphorus in milk, cereals, lecithin, etc., the following method gives good results, and has the additional advantage of enabling a simultaneous determination of total chlorine and sulphur to be made: The substance is heated with a large excess of pure fuming nitric acid containing 1 per cent. of silver nitrate, which effects a rapid destruction of organic matter. The silver chloride formed is separated, and the excess of silver in the filtrate precipitated with hydrochloric acid. The phosphoric acid can then be precipitated from this filtrate by means of ammonium molybdate. When the sulphur is to be determined the bulk of the nitric acid should first be removed by evaporation. C. A. M.

ORGANIC ANALYSIS.

On Nicloux's Method of determining Alcohol. M. E. Pozzi-Escot. (*Ann. de Chim. anal.*, 1902, vii., 11, 12.)—Nicloux's method is a colorimetric one based upon the reducing action of alcohol upon potassium bichromate in an acid solution (ANALYST, xxii., 161 and 263).

The author has made a series of experiments with this method, and finds that all the alcohols met with in fermented liquids behave like ethyl alcohol towards the reagent, and that aldehyde, contrary to Nicloux's assertion, has also a reducing action.

In order to obtain comparable results, even with absolutely pure ethyl alcohol, it is essential to maintain absolutely identical conditions, and even then the author has obtained discordant figures in duplicate experiments. C. A. M.

Detection and Estimation of Anthranilic Methyl Ester. E. Erdmann. (*D. Chem. Ges. Ber.*, 1902, xxxv., 24; through *Chem. Zeit. Rep.*, 1902, 40.)—This ester has been found in essential oils, and is best determined by the use of such reagents as give azo colouring matters with primary aromatic amines. Any colouring matter soluble in water may be employed—*e.g.*, the combination with β-naphthol-disulphonic acid R—and the operation can be made quantitative by colorimetry. If, however, only small quantities of the ester are present, the author prefers to dilute the diazotized solution to a known volume, and then to titrate it with an alcoholic solution of β-naphthol. The colouring matter thus falls in an insoluble condition, and the end-point of the titration may be ascertained either by "spot" tests or by filtering off a little of the liquid and trying it with both the diazo compound and with naphthol. The precipitate is orange-coloured, dissolving in strong sulphuric acid to a red-violet. Even a few milligrammes of anthranilic methyl ester may be determined in this manner, and the presence of the corresponding compound of methylanthranilic acid does not interfere. F. H. L.

A Modification of Zeisel's Method for the Estimation of Methoxyl Groups. J. T. Hewitt and T. S. Moore. (*Proc. Chem. Soc.*, xviii., 10.)—The authors describe a modification of Zeisel's method, in which the condenser supplied with water at 40° is replaced by a fractionating column. The potash bulbs, containing water and red phosphorus, are dispensed with, and the time taken in fitting up the apparatus and carrying out the operation is much less than in Zeisel's original method. A. G. L.

On the Determination of the Methoxyl-Group in Substances containing Sulphur. Felix Kaufler. (*Monatshefte f. Chem.*, xxii., 1105.)—It is well known that Zeisel's method yields low results when applied to substances containing sulphur. The following method is applicable to those cases in which the methoxyl-group can be removed by saponification—that is, to the esters of methyl alcohol: The substance and 3 to 6 c.c. of solution of KHO (specific gravity 1·27) are placed in a small distilling flask, the side-tube of which communicates with a U-tube containing pieces of pumice heated after soaking in copper sulphate solution, which serves to dry the methyl alcohol. The other end of the U-tube is fitted to a Winkler absorption flask by means of some arrangement which prevents the passing back of the liquid. This flask contains hydriodic acid (specific gravity 1·7), and is connected to the Zeisel apparatus. During the determination a current of purified air is drawn through the whole apparatus, and two small distilling flasks are used as receivers for the methyl iodide, the side-tube of the second of the flasks being connected with the pump.

At the commencement of the operation the air current is started, the U-tube warmed to 80° to 90°, and the Winkler flask cooled by a mixture of ice and salt. The flask containing the substance is then slowly heated, by means of an oil-bath, until a gentle boiling commences, which is allowed to continue until the contents of the flask are dry, or nearly so. The flask is then allowed to cool, a further quantity of solution of KHO introduced, and the heating renewed until the contents of the flask are again dry. The freezing mixture is then removed from the Winkler flask, which is allowed to stand at the room temperature for half an hour, after which it is carefully heated to 140° to 150°, and kept at this temperature until the methyl iodide has been completely distilled off, the current of air being continuously maintained. Up to this point the determination requires three to four hours; the remainder of the process is exactly as in Zeisel's method.

From the test analyses it appears that the results furnished by the method are satisfactory for all practical purposes. In conjunction with Zeisel's method it might be used to differentiate between saponifiable and non-saponifiable methoxyl-groups.

A. G. L.

The Adulteration of Turpentine with "White Spirit." A. and P. Andouard. (*Journ. Pharm. Chim.*, 1902, xv., 99-101.)—The authors state that the adulteration of turpentine with light petroleum oil is now very prevalent in France, a special product being imported from America for the purpose under the name of "White Spirit."

A specimen of this substance examined by the authors had the following characteristics: Colour, violet-blue fluorescence; specific gravity at 15° C., 0·807; rotation in 200-millimetre tube, – 1·2°; boiling-point, 150° to 160° C., leaving a residue of 42 per cent. beginning to boil at 205° C., and having a rotation of – 0·2°.

The following results were obtained with a sample of pure turpentine and with six commercial samples thus adulterated:

	Specific Gravity at 15° C.	Residue at 205° C. Per Cent.	Rotation of Original Substance.	Rotation of Residue.
Pure turpentine	0·871	6	– 63·1	– 6·4°
Turpentine adulterated with white spirit	0·860 to 0·867	16 to 21	– 52·2 to 57·3	– 8·0° to 9·6°

The authors call attention to the curious fact that the optical rotation of the residue from the adulterated samples was higher than that obtained from the pure turpentine, whilst the contrary was to have been expected.

For a quantitative determination of the added substance, they recommend the destruction of the turpentine hydrocarbons by means of fuming nitric acid, which leaves the mineral oil practically unaltered.

C. A. M.

Chemical Composition of Norwegian Wood Tar. J. A. Mjoen. (*Zeits. f. angew. Chem.*, xv., 97.)—A. The retort tar is made in factories from the sawdust of pine-wood together with some fir-wood. These are soaked in water for some time, then dried. The tar is distilled off in the absence of air, and is a by-product in the manufacture of wood vinegar. The tar is brownish-black to black in colour, has an acid reaction, a penetrating smell, and a specific gravity of 1·07.

B. In the manufacture of charcoal-burner's tar the wood is partially burnt in a furnace through which a current of air passes. Consequently a large portion of the volatile constituents is lost. In colour it resembles the retort tar. It has a weak acid reaction, an aromatic smell, and a specific gravity of 1·06. In consistency it is thicker than retort tar.

	A. Norwegian Retort Tar made from Sawdust of Firs and Pines.		B. Charcoal Burner's Tar from Gudbrangdalen.		Beechwood Tar from Austria.		Bohemian Pine Tar.	
Aqueous solution ...	—	4·45%	—	5·0%	—	7·2% strongly acid	—	3·7%
Acetone	0·4%	—	0·07%	—	—	—	—	—
Total aldehydes and ketones	—	0·8%	—	0·5%	—	—	—	—
Acetic acid	0·36%	—	0·37%	—	—	—	—	—
Total fatty acids	acids detected: acetic, propionic, butyric, valeric	3·64%	—	1·78%	—	—	—	—
Guaiacol and creosote ...	creosote about 20%	—	creosote not determined	—	guaiacol 10·5%	—	creosote about 7·5%	—
Total phenols and phenol derivatives	—	44·7%	—	21·5%	—	41·0%	—	17·0%
Retene	not detected	—	4·5%	—	—	—	—	—
Pitch	—	20·6%	—	10·1%	—	21·0%	—	29·0%
Total hydrocarbons ...	—	22·5%	of these about 20% aliphatic, 80% aromatic	46·0%	—	18·0%	—	40·0%
Esters	—	—	1·4%	—	—	—	—	—
Alcohols	methyl alcohol detected: allyl alcohol	about 1·0%	—	—	—	—	—	—

The author's object was to ascertain the actual composition of the tars, not what substances could be obtained from them. Care was therefore taken to protect them as far as possible from the action of heat, air, and light.

The tar was distilled under reduced pressure, the distillate being divided into two portions—light and heavy wood oil. From each portion some aqueous solution separated. In that which separated from the light oil acetone was determined by conversion into iodoform and weighing. The iodoform obtainable from the other aqueous solution was similarly determined, and the aldehydes and ketones in the oils were estimated by known methods. The acetic acid was determined by titrating another portion of the first aqueous liquid with standard caustic soda. The phenols and the fatty acids were extracted from the different fractions by appropriate treatment with various bases. After the aldehydes, ketones, fatty acids, and phenols had been removed, the hydrocarbons and other neutral bodies remaining were washed with water and weighed. The esters were determined in these by saponification with alcoholic potash, evaporating, dissolving in water, acidifying, and weighing the acids precipitated. The creosote was apparently determined by fractional distillation at atmospheric pressure, the portion coming over between 200° and 220° C. being collected and weighed. A. M.

On the Use of Millon's Reagent. O. Nasse. (*Pflüger's Archiv*, 1901, lxxxiii., 361-368; through *Zeit. für Untersuch. der Nahr. und Genussmittel*, 1902, v., 14-15.)—The author uses an aqueous solution of mercuric acetate instead of mercuric nitrate, adding a few drops of a 1 per cent. solution of sodium or potassium nitrite. The solution is usually sufficiently acid in itself, but a little dilute acetic acid may be added if necessary. The presence of nitric acid is unnecessary, and the action is to be considered a nitroso-reaction rather than a nitration. The reaction is retarded by hydrogen peroxide, by the presence of too much chloride, and by alcohol, which latter, however, can be added after cooling without influence. With the simple hydroxylated benzene derivatives, and those in which only one hydrogen atom has been substituted, the reagent as prepared by the author gives different colorations. The ortho compounds yield a brown-red colour, the para compounds blue-red to blue, and the meta compounds various tints. Albuminous bodies and their decomposition products, tyrosin, and the derivatives of *p*-cresol give the blue-violet para reaction. Almost all phenols and salicylic acid give the para reaction. The conversion of ortho into para oxybenzoic acid by fusion with potassium hydroxide can be shown by the reagent, the former being coloured red-brown, and the latter blue-red.

The phenol-aldehyde reaction has been recommended by Denigès (*Compt. Rendu*, 1900, cxxx., 583) for the detection of tyrosin, and the author finds that when this body is gently warmed with 2 to 3 c.c of a mixture of concentrated sulphuric acid with a few drops of formaldehyde, the solution is coloured red-brown, which changes to green on the addition of glacial acetic acid. The three hydroxybenzoic acids give a similar red-brown colour, but these are not turned green by glacial acetic acid.

W. P. S.

INORGANIC ANALYSIS.

Assay of Zinc Precipitates for Gold and Silver. C. Fulton and C. Crawford. (*Eng. and Mining Journ.*, 1901, lxxii., 813; through *Chem. Zeit. Rep.*, 1902, 13.)—Ordinary assay processes are useless here, much of the gold and silver being lost by volatilization and in the slag. The crucible test gives the highest average results for gold, but not for silver, and the loss in the slag varies even when the same conditions are maintained. The best proportions for the crucible test are: 1 to 10 assay-tons of zinc precipitate, 70 grammes of litharge, 5 grammes of sodium carbonate, 1 gramme of fluorspar, 5 grammes of quartz, and 2 grammes of borax. Of combined wet and dry methods, extraction with nitric acid is not satisfactory, owing to dissolution of gold. The authors, therefore, recommend the following: 1 to 10 assay-tons of the sample are boiled for one hour with 20 c.c. of strong sulphuric acid and 60 c.c. of water, cooled, and diluted to 100 c.c.; 75 c.c. of a solution of rock-salt and 20 c.c. of one of lead acetate are added, and the precipitate, after settling for one hour, is collected and dried. This, with the filter ash, is cupelled with 30 or 40 grammes of lead. The results are good and very uniform: the gold is a little lower than that given by the crucible process, but the silver is higher. The loss in the slag is relatively small; but this slag and the cupel may be examined subsequently, and the necessary correction made. The sulphuric acid must be free from nitric acid.

F. H. L.

Volumetric Estimation of Bismuth in Ores. A. W. Warwick and T. D. Kyle. (*Eng. and Mining Journ.*, April 13, 1901; through *Chem. News*, 1902, lxxxv., 3.)—One gramme of the finely-powdered ore is evaporated to dryness with 5 or 10 c.c. of strong nitric acid; another 5 c.c. of acid and 25 c.c. of water are added, and the whole is diluted to 100 c.c. Five grammes of ammonium oxalate or oxalic acid are introduced, boiled for five minutes, allowed to settle, and the supernatant liquid filtered off. The precipitate is boiled twice with 50 c.c. of water, and the washings are passed through the same paper. With an ordinary 10 per cent. ore this treatment should suffice to convert the bismuth oxalate into the basic salt; but if the filtrate is still acid, boiling must be repeated to neutrality. The precipitate on the paper is then dissolved in 2 to 5 c.c. of 1 : 1 hydrochloric acid, receiving the liquid in the beaker containing the bulk of the basic oxalate; this is warmed till entirely dissolved, and then diluted to 250 c.c. with hot water. The solution is neutralized with ammonia, and the resulting precipitate taken up in 1 : 4 sulphuric acid, adding a few c.c. in excess. Finally the liquid is titrated at between 70° and 100° C. with permanganate. A permanganate solution in which 1 c.c. = 0·010 gramme Fe, will be equal to 0·0186 gramme bismuth; by diluting 100 c.c. of this with 86 c.c. of water a solution of permanganate will be obtained, of which 1 c.c. should equal 0·010 gramme of bismuth. A permanganate solution 1 c.c. = 0·010 gramme Fe; found = 0·01868 gramme Bi. 100 c.c. permanganate above + 86 c.c. of water; 1 c.c. found = 0·01017 gramme Bi. Lead, iron, copper, zinc, arsenic, and tellurium do not interfere with the process. Care must be taken to avoid using too little or too much nitric acid. Hydrochloric acid must not be used to dissolve the ore. The results are accurate enough for all commercial work, and an analysis can be carried out in thirty or forty minutes. The figures quoted show maximum errors of − 0·0032 and + 0·001 gramme in determining 0·5 gramme of bismuth in presence of lead, copper, zinc, iron, and arsenic.

F. H. L.

Precipitation of Ferrous Sulphide by Means of Sulphuretted Hydrogen. A. Coppadoro. (*Gazz. Chim. Ital.*, 1901, xxxi. [2], 217; through *Chem. Zeit. Rep.*, 1902, 40.)—The author points out that when sulphuretted hydrogen is led through a ferric solution, the sulphur which is deposited carries down with it a small quantity of ferrous sulphide, as may be shown by extracting the precipitate with carbon bisulphide. A small amount of ferrous sulphide is also obtained when a solution containing sodium thiosulphate and ferric chloride is treated with an acid. The indispensable condition for the formation of the ferrous sulphide is the simultaneous separation of sulphur.

F. H. L.

Action of Certain Reagents on Free Silicon. P. Lebeau. (*Bull. Soc. Chim.*, 02, xxvii., 42-44.)—A series of experiments are described in detail, which show that e reagents usually employed to dissolve steel, etc., are without action upon free licon, even when in a fine state of division, and that that element will be left in the dissolved residue. C. A. M.

The Determination of Persulphates. Charles A. Peters and Seth E. oody. (*Zeits. anorg. Chem.*, xxix., 326.)—The authors have been induced to ake a comparison of the various methods for the estimation of persulphates by the pearance of a paper by Namias (abstract, *Chem. Centrbl.*, 1900, ii., 806), in which e method of Le Blanc and Eckardt (*Zeitschr. Electrochem.*, v., 355) is stated to be accurate. In this method the solution of the persulphate is treated with an excess a standard solution of ammonium ferrous sulphate, either for ten minutes at temperature of 60° to 80°, or for eleven hours at room temperature, and the excess ferrous salt titrated with standard permanganate solution. Both modifications of e method gave accurate and concordant results, and experimentally it was found necessary to replace the air in the titrating vessel by carbon dioxide.

In Grützner's method (*Chem. Centrbl.*, 1900, i., 835) the solution of the per-lphate is heated to boiling with a standard solution of arsenious acid, and potassium sodium hydroxide, cooled, acidulated with sulphuric acid, and made alkaline with tassium bicarbonate; the excess of arsenious acid is then determined with a andard iodine solution. This method also was found to give reliable results, ovided that the small amount of arsenious acid oxidized by the reagents added was termined, and this correction applied in calculating the results.

In Mondolfo's method (*Chem. Zeit.*, xxiii., 699) the persulphate solution is ated with potassium iodide in a closed bottle in a water-bath to 60° to 80° for a ort time, and the liberated iodine titrated with thiosulphate. There appeared to a tendency towards low results with this method, but good results were obtained en the volume was kept small (25 c.c.) and the time of heating increased up to irty minutes (for 0·12 gramme persulphate).

The method described by Namias (*loc. cit.*) differs from Mondolfo's only in the ct that the mixture of persulphate and iodide is allowed to stand for eleven hours room temperature instead of being heated for a short time. This method also s found to give low results unless the volume of the solution is kept small. In ither of the two last methods is the addition of sulphuric acid advantageous.

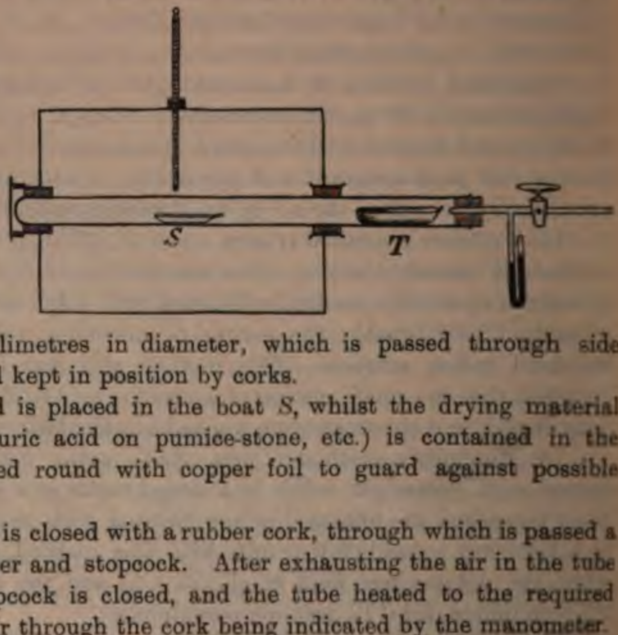
Gooch and Smith's method (*Journ. Sci.* [*Sill.*], 42, 220) for the estimation of lorates was also used for persulphates as follows: To the solution of the persulphate excess of a standard solution of potassium iodide and an excess of acid potassium senate are added, the whole made strongly acid with sulphuric acid, diluted, and iled down to a third of its volume; after cooling, the solution is made alkaline with tassium bicarbonate, and the residual arsenite titrated with iodine. The difference tween the amount of iodine required and the amount contained in the potassium lide added originally corresponds to the persulphate present. The results obtained re accurate, and agreed well with those obtained by the other methods.

In conclusion, the method of Le Blanc and Eckardt appears to be simple, rapid, and accurate, and furnishes results lying between those given by the other methods, Mondolfo's and Namias's methods giving the lowest, and Grützner's and the arsenate method the highest results, the extreme difference being about 1·5 milligrammes on 0·12 gramme.

A. G. L.

APPARATUS.

A Simple Apparatus for Drying Substances under reduced pressure at any required Temperature. F. Pregl. (*Zeit. anal. Chem.*, 1901, xl., 781-785.)—The device shown in the figure is of use, not only for drying hygroscopic substances, but is also particularly suitable for determining the water of crystallization of crystals. As a rule, substances are dried to constant weight within half a day.



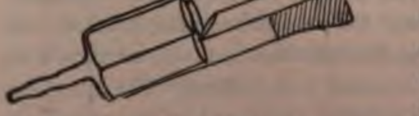
It consists of a thick glass tube about 41 centimetres in length and 20 millimetres in diameter, which is passed through side tubulures in a drying oven and kept in position by corks.

The substance to be dried is placed in the boat *S*, whilst the drying material (phosphorus pentoxide, sulphuric acid on pumice-stone, etc.) is contained in the larger boat, which is wrapped round with copper foil to guard against possible spirting.

The open end of the tube is closed with a rubber cork, through which is passed a tube with a mercury manometer and stopcock. After exhausting the air in the tube by means of a pump the stopcock is closed, and the tube heated to the required temperature, any leakage of air through the cork being indicated by the manometer.

C. A. M.

Receiver for Fractional Distillation under Diminished Pressure. W. Burstyn. (*Oesterr. Chem. Zeit.*, 1901, iv., 563.)—The three small tubes which serve as actual receivers are lightly held together by means of a rubber ring, and they are dropped into the larger tube as shown. By having the end of the condenser tube slightly bent, its orifice can be brought opposite the mouth of each of the small receivers in succession by simply rotating the outer vessel on its rubber cork. The tube at the lower end of the main vessel leads to the pump. The rubber cork should be lubricated with strong pure glycerin, which is better than



llodion, as it does not attack the rubber, has no appreciable vapour tension, and n be quickly washed off with water. F. H. L.

Laboratory Appliances. **R. Gnehm.** (*Zeits. f. angew. Chem.*, xiv., 1208.)—g. 1 shows a simple mill for pulverizing solid bodies, treating cellulose with strong da lye, etc. The iron tube *A*, about 100 millimetres in diameter, is at either end rewed to the two brass plates, C_1C_2, which are proded with extensions, D_1D_2, that act as an axle and st in the supports E_1E_2. To introduce or remove the bstance the plate C_2 is unscrewed. At the same time number of iron cylinders, *W*, or balls, *K*, are introced. The drum is turned by means of the pulley *F*.

For operations which require continual stirring, gether with heating under an inverted condenser and e introduction of liquid, the appliance shown in Fig. 2 ay be used. The stirrer is driven from the pulley *G* and rotates in the tube *F*, hich is made of glass or metal, and is fixed by means of the cork E into the pliance. A. M.

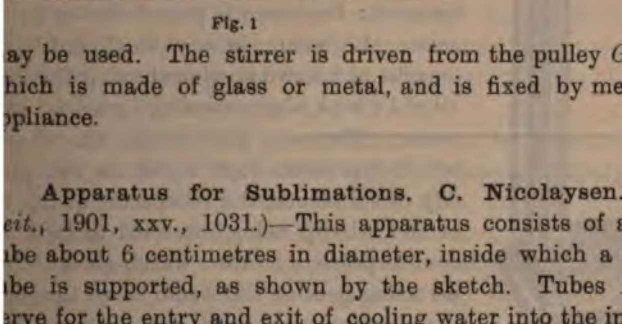


Fig. 1

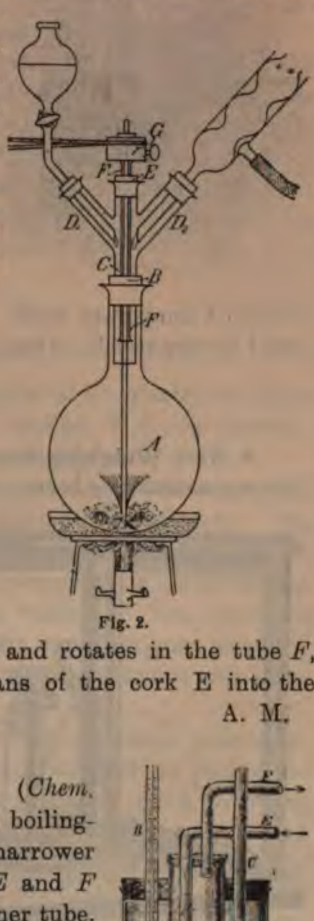
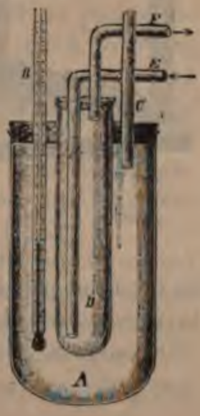


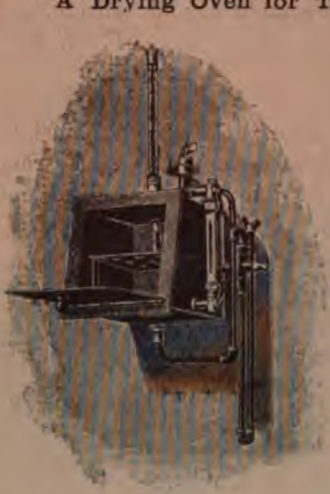
Fig. 2.

Apparatus for Sublimations. **C. Nicolaysen.** (*Chem. it.*, 1901, xxv., 1031.)—This apparatus consists of a boilingbe about 6 centimetres in diameter, inside which a narrower be is supported, as shown by the sketch. Tubes *E* and *F* rve for the entry and exit of cooling water into the inner tube. is open at both ends. Small quantities of materials can be fely deposited on the outside of *D* without danger of the blimate dropping; if it be desired to collect larger amounts, small basin might be slung under *D* so as to collect any falling atter.

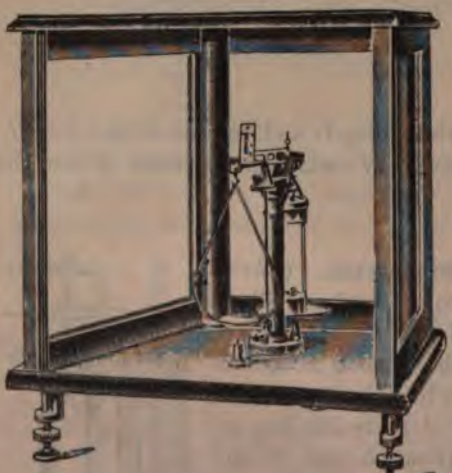
F. H. L.



A Drying Oven for Temperatures exceeding 100° C. (*Chem. Zeit.*, 1901, xxv., 1086.)—This is an apparatus which can be set to give any desired temperature up to 105° (or even 110°, if necessary), working with pure water boiling under pressure. The thermostat shown on the right of the oven in the diagram contains about 140 grammes of mercury, and is so constructed that when the temperature, and therefore pressure, rises to the predetermined point, the mercury cuts off the gas supply, leaving only a pilot flame alight. By introducing small extra quantities of mercury into the tube the maximum temperature can be lowered by a degree at a time. A brass-jacketed gauge-glass is also fitted at the side. On the top is a valve which admits air to the steam space when the oven is cold. The front slopes as shown, so that the door needs no latch or other fastening; when it is open it lies horizontally to form a temporary shelf. The oven can be affixed to the wall, or provided with a stand for the bench. It is patented by G. Christ and Co., of Berlin. F. H. L.



A New Weighing-out Balance. F. Mach. (*Chem. Zeit.*, 1901, xxv., 1139.)—This apparatus has been specially designed to economize time in the weighing off of the same quantity of material for analysis, especially when the same watch-glass, etc., is always used. It includes three special features: the beam is placed end-on in the case; the beam is a "steel-yard," the lengths of its two portions having the ratio 10 : 1; and the arresting gear is automatic. The rear pan carries the weight, which represents (ten times) the net quantity of substance, the receptacle at the top of the stirrup being intended to hold the counterpoise of the watch-glass. The arresting gear of the beam is so arranged that it carries the rear pan with counterpoise and weight; and only when one-tenth or more of the substance which has to be weighed is put into the watch-glass on the front pan does the steel-yard beam come into play. Both pans have stops to prevent oscillation. The delicacy of the balance exceeds 1 milligramme per 100 grammes. It is made by W. Spoerhase, of Giessen. F. H. L.



On the Sensitiveness of a Thermo-regulator. A. W. C. Menzies. (*Proc. hem. Soc.*, xviii., 8.)—With the arrangement described below, the temperature of the ath could be kept constant at 18° within a total range of 0·008° C. The glass horseshoe" reservoir was of the usual form, having a capacity of 390 c.c. and an uter surface of about 650 square centimetres; the ordinary U-form regulating be containing mercury was fused on to this reservoir, all the tubes containing closed liquid being placed in the water of the bath. Toluene being used for e expanding liquid, the stopcock closing one limb of the U-tube was lubricated ith syrupy phosphoric acid. The bath held 14 litres, and was of enamelled iron, ith no jacket. The stirrer was driven by a small electromotor, some of the bration being communicated to the regulator to aid the free movement of the ercury. The gas-supply was regulated so that the by-pass flame was nearly fficient to maintain the temperature. A tube of 3·1 millimetres bore was used for e outer limb of the regulator, and ordinary unfiltered coal gas was used. For arrower tubes the gas should be filtered and dried. A bore of 1·9 millimetres for e outer limb gave a temperature variation of not more than 0·0025°. A. G. L.

ANNUAL MEETING OF THE INSTITUTE OF CHEMISTRY.

HE twenty-fourth annual general meeting of the Institute of Chemistry of Great ritain and Ireland was held at 30, Bloomsbury Square, London, W.C., on Monday, arch 3, Professor J. Millar Thomson, LL.D., F.R.S., President, in the chair.

The ordinary annual business of the Institute was transacted, and the President elivered an address dealing with work of the Institute during the past year. The ll of members has increased by 36, making the total of 1,040 Fellows and Associates present on the register, while there are 130 students in course of training at the lleges and institutions recognised by the Council of the Institute. The President marked on the higher percentage of passes in the recent examinations, which he nsidered pointed to the fact that the thoroughness of the examinations was more lly realized by those preparing for them.

He also mentioned the establishment of an examination in biological chemistry, y means of which the Council hope to encourage analytical chemists to pursue the udy of bacteriology, and thus be able to deal with chemical work involving cteriological knowledge. The examination is specially suited for such as intend tering the practice of chemistry in its relationship to the chemistry and bacteriology food, water, sewage, and effluents, and the practical applications of biological emistry to such industries as brewing, tanning, etc. He called attention to the ct that the Institute held an examination in Ireland in July last, and that the ouncil hope in future to arrange for a regular examining centre in Dublin.

During the past year the Council have ceased to recognise the junior, second ass, and lower grade professional preliminary examinations of various examining odies, and they have now under reconsideration the subjects which shall be comulsory to persons intending to proceed to the examinations of the Institute.

The Council also reported to the members that during the past year they had addressed to the Local Government Board, and to various county and borough authorities, communications with reference to the combination of the two appointments of Medical Officer of Health and Public Analyst. It has been pointed out that a Public Analyst requires special training and experience in analytical chemistry far beyond that which is usually acquired by medical practitioners in the course of their professional education; also, that where the appointments are proposed to be combined, the selection of candidates for both positions is necessarily limited. The Council hold the opinion that under the provisions of the Sale of Food and Drugs Act of 1875 it was intended that the Medical Officer of Health and the Public Analyst for a borough or district should be two distinct persons, inasmuch as the Medical Officer is one of those officers empowered by the Act to submit suspected samples for analysis. The qualifications and examinations of the Institute are accepted by the Local Government Board for public analytical appointments, and it is the desire of the Council that no lower standard of chemical training and experience than that prescribed by the Institute of Chemistry should be accepted for such important and responsible public appointments as those of Public Analysts under the Sale of Food and Drugs Act.

REVIEWS.

The Composition of Dutch Butter. By Dr. J. J. L. van Rijn. London: Baillière, Tindall and Cox, 1902.

This book contains a very large number of exhaustive analyses of butter from farms in Holland, which were expected to yield abnormal figures, and they are ably discussed. Two main facts are stated: Firstly, that genuine butters may give a Reichert-Wollny figure of 19, 18, or even 17; and, secondly, that a "rise [in Reichert-Wollny figure] begins shortly after the cows have been stabled."

The first fact, which has been known for years, is liable to great misinterpretation, and more than one person has acquired the idea that "50 per cent. of all Dutch butters" fall below 25 c.c. Reichert-Wollny. Read with proper understanding, this book shows nothing of the kind, and the author would probably be the first to admit this. The second fact, which has received confirmation, points to the remedy for low Reichert-Wollny figures, and shows that, were a reasonable standard prescribed, it would be but a matter of organization to comply with it. H. D. R.

Food and Drugs. A Manual for Solicitors, Public Analysts, Inspectors, etc.; being a Consolidation of the various Food Acts. By Charles James Higginson. Second edition, revised and enlarged. London: Effingham Wilson, 11, Royal Exchange.

The first edition of this most useful little book appeared in 1900. It has met with well-deserved appreciation, as shown by the need for a second edition. This has been brought quite up to date, references to "milk-blended butter" and to the Board of Agriculture Milk Regulations being included. The list of High Court decisions is very complete, and copious and well-arranged indices render reference easy.

Among the many legal works on the Food Acts none deserves a place in the Public Analyst's library more than Mr. Higginson's. O. H.

INDEXED

The Analyst

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VOLUME XXVII.—No. 314.
MAY, 1902.

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THE ANALYST.

MAY, 1902.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE monthly meeting of the Society was held on Wednesday evening, April 9. The President (Dr. J. Augustus Voelcker, M.A., B.Sc.) occupied the chair.

The minutes of the previous meeting were read and confirmed.

Certificates of proposal for election to membership in favour of Messrs. Walter Henry Jollyman, A.I.C., John T. Norman, John Golding, F.I.C., and William Bowen, were read for the second time; and certificates in favour of Messrs. Meredith Wynter Blyth, B.A. (Cantab.), B.Sc. (London), F.I.C., Public Analyst for the Boroughs of Brighton and Eastbourne, and Walter Frederick Sutherst, Ph.D. (Geneva), A.I.C., Analyst and Lecturer in Chemistry at the Cheshire Agricultural College, Holmes Chapel, Crewe, were read for the first time.

Dr. J. J. L. van Rijn was elected a member of the Society.

The following papers were read: "Alkaline Waters from the Lower Greensand," by W. W. Fisher, M.A.; "The Rapid Estimation of Boric Acid in Butter," by H. Droop Richmond and J. B. P. Harrison; "The Detection and Estimation of Minute Quantities of Sulphuretted Hydrogen in Coal Gas," by W. J. Dibdin and R. G. Grimwood; "The Hexabromides of Glycerides and Fatty Acids," by John Walker and George Warburton; and a "Note on the Estimation of Mineral Oil in Rosin Oil," by John Walker and Charles D. Robertshaw.

ON THE INDIRECT ESTIMATION OF ALKALIES IN WATERS.

BY W. W. FISHER, M.A., F.I.C.

(*Read at the Meeting, March* 5, 1902.)

IN the analysis of waters where mineral constituents have to be estimated, the more abundant bases determined are the lime, magnesia, and soda (inclusive of potash), and the acid radicles are usually chlorine, sulphuric acid, carbonic acids, and possibly nitric acid. After such determinations have been made, it becomes a somewhat tedious task to calculate the quantities of the compounds present, and much arithmetical work is involved in finding out whether the estimations duly balance each other. But this work of computation is greatly simplified if the relative molecular proportions of the several acids and bases are first obtained by dividing the quantities of each by their molecular weights, and taking the totals of acids and bases separately. If the work is absolutely correct, the totals will be alike, and they will be always near each other if the determinations are good; while any marked difference would indicate some analytical error, as only neutral compounds are present in the total solid residue.

The following analysis will serve as an illustration:

	Grains per Gallon.	Molecular Weights.	Molecular Proportions.
Total solids dried at 100° C.	243·00		
Total solids dried at 240° C.	230·00		
Soda, Na_2O	53·14	÷ 62	0·8571
Magnesia, MgO	11·99	÷ 40	0·299
Lime, CaO	35·28	÷ 56	0·630
Total of bases	100·41		1·7861
Chlorine, Cl_2	18·00	÷ 71	0·2535
Sulphuric acid, SO_3	103·88	÷ 80	1·2985
Carbonic acid, CO_2	9·55	÷ 44	0·2170
Nitric acid, N_2O_5	0·84	÷ 108	0·0077
Total of acids	132·27		1·7767
Deduct oxygen = Cl_2	4·07		
	128·20		
Total of salts	228·58		
Silica, etc. (difference)	1·42		
Total solids	230·00		

The subsequent work of combining the acids and bases, apportioning to each its due quantity, becomes very simple when the molecular proportions are employed, as 0·2535 molecules of chlorine will require 0·2535 molecules of sodium to form sodium chloride, no correction for the equivalent oxygen being required; and after the nitric acid the remaining soda—viz., 0·5959—will neutralize an equal number of sulphuric acid molecules, and so on till all the salts are obtained.

	Molecules.
(2) NaCl	0·2535 × 117 = 29·66
(2) $NaNO_3$	0·0077 × 170 = 1·31
Na_2SO_4	0·5959 × 142 = 84·62
$MgSO_4$	0·299 × 120 = 35·88
$CaSO_4$	0·4036 × 136 = 54·89
$CaCO_3$	0·2264 × 100 = 22·64
Total of salts	229·00

The molecular proportions may also be used with signal advantage for the indirect valuation of alkalies. The direct estimation of soda in waters is a troublesome and lengthy operation, and in technical analyses is frequently omitted. Yet a knowledge of the amount of alkali in waters is often very desirable in order to allow the amounts of the salts to be approximately stated. It is generally impracticable to arrive at the amount of alkali by deducting the other constituents from the total solid residue, because many residues rich in sulphates retain water in variable quantity according to the conditions of drying.

But when the lime and magnesia are known, and also chlorine, sulphuric, and carbonic acids (and possibly nitric acid also), the difference between the molecular totals of acids and bases gives a close approximation to the amount of alkali.

In the analysis quoted above the total acids being 1·7837 molecules the bases would be the same, from which, if we deduct 0·929 molecules of lime and magnesia, the residue of 0·8547 is soda (inclusive, of course, of traces of potash). Further multiplying 0·8547 by 62, we get 52·99 grains per gallon, which is little below the actual determination.

The result may be controlled by a direct estimation of the total bases as sulphates.

The following example shows how an indirect determination works out:

Wheatfield water.
Total solids 79·24 at 100° C. At 140° 78·12.

	Grains per Gallon.		Molecular Weights.	Molecular Proportions.
Cl	9·70	÷	71	0·1366
SO_3	4·66	÷	80	0·0582
CO_2	21·56	÷	44	0·4900
				0·6848
Na_2O by difference	[40·12]	÷	62	[0·6473]
MgO	0·5	÷	40	0·0125
CaO	1·4	÷	56	0·0250
				0·6848

	Molecules.	Grains.		
(2) NaCl	0·1366 × 117 =	15·98	Salts ...	75·75
Na_2SO_4	0·0582 × 142 =	8·26	SiO_2 ...	2·5
Na_2CO_3	0·4525 × 106 =	47·96	Fe_2O_3 ...	0·84
$MgCO_3$	0·0125 × 84 =	1·05		
$CaCO_3$	0·025 × 100 =	2·5		79·09
Total salts		75·75		

ON OXIDIZED OILS.

By J. Lewkowitsch, Ph.D.

(*Read at the Meeting, March* 5, 1902.)

In my last paper, read before this Society at a meeting, November 1, 1899,* I pointed out that the examination of the "oxidized acids" occurring in blown oils and solid linseed oil had been taken in hand by me.

This investigation has made very slow progress, and I should not have published the following incomplete results had not a paper appeared by Em. Lecocq and

---

* Analyst, 1899, p. 319.

TABLE I.—*Blown Oils.*

	I.	II.	III.	IV.	V.	VI.	VII.	VIII.	IX.	X.	XI.	XII.	XIII.	XIV.	XV.
	Acid Value.	Sap. Value.	II.-I.	Iodine Value.	Total Soluble Acids.	Specific Gravity.	Unsa-ponifi-able.	Oxidized Acids.	Hehner Value.	Acetyl Value.		Sap. Value of Acetyl-ated Oil.	True Acetyl Value × 0·55.	Hehner Value after Acetyla-tion.	XII.-II.
										Ap-parent.	True.				
	Mgrms. KOH.	Mgrms. KOH.	Mgrms. KOH.	Per Cent.	Mgrms. KOH.		Per Cent.	Per Cent.		Mgrms. KOH.	Mgrms. KOH.	Mgrms. KOH.			
Ravison rape...	10·47	198·31	187·84	72·66	35·89	0·9685	1·23	21·22	83·52	88·37	52·93	243·2	29·11	—	44·9
East India rape	13·25	215·57	202·32	61·92	56·26	0·9623	0·93	20·74	82·18	102·87	46·61	253·33	25·63	—	37·76
Cottonseed ...	9·41	224·59	215·18	65·74	46·49	0·9785	1·37	29·39	82·59	110·73	64·29	273·30	35·36	83·85	48·71
Solid linseed oil (linoleum mass)	*	287·47	—	52·2	136·9	—	1·33	53·01	53·92	—	—	367·75	—	—	—
Maize ...	7·33	208·63	201·30	90·7	49·13	0·9806	2·28	31·93	82·34	113·16	63·37	268·75	34·85	—	60·1

* Acid value was difficult to take. Experiments made by adding excess of alkalies and titrating back led to the impossible value 369·9 (?).

TABLE II.—*Total Fatty Acids.*

	I.	II.	III.	IV.	V.	VI.	VII.	VIII.	IX.	X.	XI.
	Acid Value.	Sap. Value.	II.-I.	Iodine Value.	Total Soluble Acids.	Hehner Value.	Acetyl Value.		Sap. Value of Acetyl-ated Acids.	IX.-II.	Hehner Value of the Acetyl-ated Acids.
							Ap-parent.	True.			
	Mgrms. KOH.	Mgrms. KOH.	Mgrms. KOH.	Per Cent.	Mgrms. KOH.		Mgrms. KOH.	Mgrms. KOH.	Mgrms. KOH.		Per Cent.
Blown Ravison rape oil	175·14	191·7	16·56	73·31	7·26	—	50·0	42·75	227·4	35·7	—
Blown East India rape oil	171·93	190·0	18·07	60·80	10·71	—	66·2	55·5	237·8	47·8	—
Blown cottonseed oil	194·79	210·46	15·67	72·43	12·94	93·76	67·35	55·67	254·8	44·4	92·11
Solid linseed oil (linoleum mass)	209·63	248·74	39·11	60·27	59·57	81·32	115·01	55·04	304·24	55·5	84·4
Blown maize oil	192·8	209·93	17·13	88·08	29·45	86·4	88·97	59·52	267·3	57·37	—

TABLE III.—*Oxidized Acids.*

	I.	II.	III.	IV.	V.	VI.	VII.	VIII.	IX.	X.	XI.	XII.
	Acid Value.	Sap. Value.	II.-I.	Iodine Value.	Total Soluble Acids.	Hehner Value.	Acetyl Value.		Sap. Value of Acetylated Acids.	IX.-II.	True Acetyl Value × 0·55.	Hehner Value of Acetylated Acids.
							Apparent.	True.				
	Mgrms. KOH.	Mgrms. KOH.	Mgrms. KOH.	Per Cent.	Mrgms. KOH.		Mgrms. KOH.	Mgrms. KOH.	Mgrms. KOH.			
Blown Ravison rape oil	171·5	208·0	36·5	49·14	22·56	—	102·5	80·0	307·5	99·5	44·0	—
Blown East India rape oil	173·3	211·3	38·0	39·79	22·35		128·0	105·65	315·9	104·6	58·1	—
Blown cottonseed oil	174·7	220·71	46·01	48·6	36·12	—	154·4	118·28	322·69	101·98	65·02	83·85
Solid linseed oil (linoleum mass) ...	—	—	—	46·49	59·68	—	164·67	104·99	341·43	86·29	57·74	76·38
Blown maize oil	171·94	215·74	43·60	70·87	48·0	93·53	173·58	126·68	326·45	111·11	69·67	—

TABLE IV.—*Fatty Acids freed from Oxidized Acids.*

	I.	II.	III.	IV.	V.	VI.	VII.	VIII.	IX.	X.	XI.	XII.	XIII.
	Acid Value.	Sap. Value.	II.-I.	Iodine Value.	Total Soluble Acids.	Hehner Value.	Soluble Acids.	Acetyl Value.		Sap. Value of Acetylated Acids.	X.-II.	True Acetyl Value × 0·55.	Hehner Value of Acetylated Acids.
								Apparent.	True.				
	Mgrms. KOH.	Mgrms. KOH.	Mgrms. KOH.	Per Cent.	Mgrms. KOH.	Per Cent.	Mgrms. KOH.	Mgrms. KOH.	Mgrms. KOH.	Mgrms. KOH.			
Blown Ravison rape oil ...	176·8	188·6	11·8	61·88	6·97	—	6·75	42·5	35·53	220·3	31·7	19·54	—
Blown East India rape oil ...	166·6	176·8	10·2	55·93	10·09	—	8·85	47·13	37·54	219·2	42·95	20·64	—
Blown cottonseed oil ...	188·0	196·15	8·15	56·02	11·0	—	7·27	33·69	22·69	232·0	33·6	12·49	96·17
Solid linseed oil (linoleum mass)	179·97	187·58	7·61	61·31	18·89	—	—	50·25	31·36	246·11	58·53	17·24	96·05
Blown maize oil	172·37	177·68	5·31	85·52	6·14	85·54	7·54	43·8	36·7	228·76	50·52	20·18	—

H. Dandervoort (*Chem. Revue*, 1902, 13), which partly covers the same subject, although it refers to blown colza oil only.

The oils I chose for examination were: Commercial blown Ravison oil, commercial blown East India rape oil, commercial blown cottonseed oil, solid linseed oil (so-called "scrim" oil), and blown maize oil.

All the values that have been determined in these oils are set out in Table No. I., headed "Blown Oils" (see p. 140).

For the preparation of the oxidized acids, 100 grammes were saponified in the usual manner, the total fatty acids separated, and the separation of the oxidized acids from the other acids effected by means of petroleum ether.

The total fatty acids as well as their components—viz., the oxidized acids and the acids freed from oxidized acids—were examined, and the values so obtained are set out in Tables II., III., and IV. (see p. 141).

On acetylating the *original oils*, it was found that emulsions were formed in the washing-out process in the case of blown Ravison oil, rape, cottonseed oil, and maize oil. This has been pointed out already by Archbutt, but the separation caused little trouble. The blown Ravison oil formed the strongest emulsion; the scrim oil caused more trouble, the acetylated product separating as a sticky mass denser than water.

In contradistinction to the behaviour of the oils, the total fatty acids, which were also acetylated, did not give emulsions, as has also been pointed out by Archbutt.

Oxidized Acids.—The values obtained on examination are set out in Table No. III. The acetyl values were determined by the filtration method. As found before in the case of the oxidized acids from solid linseed oil, all the oxidized acids showed considerably higher saponification values than acid values. The surmise that these acids would be saturated acids is not borne out by experiment, for the iodine values are comparatively high. A difficulty arose in the determination of the iodine values, inasmuch as the oxidized acids were found to be practically insoluble in carbon tetrachloride, hence strong alcohol was used as a solvent. The error, if any, introduced thereby cannot have been a considerable one, for while a blank test with carbon tetrachloride required 50·6 c.c. of thiosulphate, the alcoholic solution took 50·00 c.c.

To obtain the true acetyl value the total soluble acids had to be determined. Naturally one would have expected to find no soluble acids, as they should all have been washed away or dissolved out in the treatment with petroleum spirit. However, considerable quantities were found in each case. The explanation suggested itself that these soluble acids were formed on treatment with the alcoholic potash by the action of the latter on the oxidized acids, for the soluble acids were determined after the original substance had been boiled with excess of alcoholic potash for the determination of the saponification value.

If this explanation be the true one, then a check experiment with the oxidized fatty acids as they were obtained originally should give no soluble acids on washing, but the actual experiment gave considerable amounts of soluble acids, in some cases almost approaching those obtained after treatment with alcoholic potash. This is shown in the following table:

SOLUBLE ACIDS IN OXIDIZED ACIDS.

Oil.	After boiling with Alcoholic KOH.	By washing with Water.
Blown Ravison rape	22·56	21·38
,, East India rape	22·35	9·5
,, cottonseed I.	36·12	16·36
,, ,, II.	26·30	21·39
Solid linseed	59·68	24·11
Blown maize	46·90	19·20

No such differences were observed in the cases of the total fatty acids and the fatty acids freed from oxidized acids, as will be seen from the following table:

SOLUBLE ACIDS IN

OIL.	TOTAL FATTY ACIDS.		FATTY ACIDS FREED FROM OXIDIZED ACIDS.	
	After boiling with Alcoholic KOH.	By washing with Water.	After boiling with Alcoholic KOH.	By washing with Water.
Blown Ravison rape ...	7·26	7·09	6·97	6·75
,, East India rape ...	10·71	9·84	10·09	8·85
,, cottonseed	12·94	12·99	11·0	7·27
,, maize	29·4	24·8	6·08	7·54

Unfortunately, the soluble acids in the solidified linseed oil oxidized acids have not been determined, as it was taken for granted at the time that they were free from soluble acids, which now appears to be very doubtful.

The values obtained by multiplying the true acetyl values by 0·55 are again remarkably low, as they should have been, of course, 100 per cent. This proves that the molecular weight of 300 is not to be taken without further evidence.

The differences between the saponification and acid values point to the presence of lactones. Further investigation is in hand, but so far it appears pretty certain that the oxidized acids are not insoluble in water, and it is not unlikely that they split off volatile fatty acids when treated in a current of steam.

Acids freed from Oxidized Acids.—It was to be expected that these acids would have no acetyl value. If such a one were obtained, it would perhaps point to the presence of hydroxy-acids, which are soluble in petroleum ether. The considerable acetyl values found and set out in Table IV. may be due to oxidation having taken place after isolation, although free access of air had been guarded against. The acids still possessed considerable iodine values, and the differences between saponification and acid values again point to the presence of lactones, although their proportion can be only very small, not amounting to more than 4 to 6 per cent. Even these acids contain small amounts of soluble acid, as shown in the table.

Further examination must show whether, under the conditions I worked with, the oxidized acids are completely insoluble in petroleum ether, for small experiments seem to show that the petroleum spirit solution of the fatty acids does dissolve some oxidized fatty acids.

The further examination of the oxidized acids is in hand, and experiments must show whether the preparation of their esters or of salts will give an insight into their nature.

As evidenced by the figures given in the table headed "Oxidized Acids," they must contain lactonic substances, since otherwise the differences between the acid and saponification values would not be explicable. If that difference be due to lactones, then it should be possible to separate them in the usual manner—viz., by neutralizing the acids with aqueous potash and extracting the solution with ether. The supposed lactones so obtained were very thick, viscous liquids, soluble in alcohol, with the exception of those lactones that were obtained from solidified linseed oil.

The saponification values of these lactones are given in the following table, Column I. The soap solution was decomposed by mineral acid, and the acid values of the recovered fatty matter determined. No lactone had been reformed, because on boiling with alcoholic potash no further amount of alkali was absorbed:

Lactones from:	Saponification Value. I.	Acid Value of Fatty Acids recovered from the Saponified Lactones. II.
Blown Ravison rape	117·8	125·3
,, East India rape	42·72	80·25
,, cottonseed	72·116	86·6
Solid linseed	35·51	57·44
Blown maize	101·1	106·9

The aqueous soap solution of the oxidized acids after the removal of the lactones was treated with mineral acid under ether, so that the separating fatty acids were immediately dissolved.

The following table shows that in the fatty matter so obtained some lactonic substances had been formed again, as evidenced by Columns I. and II.:

OXIDIZED ACIDS FREED FROM LACTONES.

	Acid Value. I.	Saponification Value. II.	Fatty Matter recovered from Soap Solution, obtained sub. II.		
			Acid Value. III.	Saponification Value. IV.	Difference. V.
Blown Ravison rape ...	172·2—175·6	220·6—221·5	184·8	223·7	38·9
,, East India rape	194·0—195·4	237·7—247·6	198·5	234·1	35·6
,, cottonseed ...	177·9—204·5	215·1—222·9	190·2	221·7	31·5
Solid linseed	190·8—194·5	255·5	212·9	249·8	37·9
Blown maize	182·2—284·3	216·0—217·7	185·8	218·8	33·0

The solutions were very dark, so that it was very difficult to titrate accurately, ence he somewhat considerable differences between duplicate determinations.

The solutions containing the completely saponified mass were treated with mineral acid, and the fatty matter so recovered again examined for acid and saponification values, with the results set out in Columns III. and IV. of the last table. Allowing for the errors caused by the very dark solution, the numbers given under Columns III. and IV. may perhaps be looked upon as giving practically the same values as those stated in Columns I. and II., with the exception of solid linseed oil. The practically constant differences in Column V. are notable.

The process of re-extracting the lactonic substances, etc., was repeated once or twice, but lactonic substances were always formed again.

My thanks are due to Messrs. C. D. Robertshaw and George Warburton for the numerous analyses made in the preparation of this paper.

DISCUSSION.

Mr. JENKINS said that in dealing with blown cotton oil of about 0·975 specific gravity he had found that the fatty acids from such oil were distinctly more soluble in methylated ether than in petroleum spirit, which appeared to confirm the author's experience. It was a rather peculiar fact that the blowing of these oils decreased their miscibility with petroleum compounds. The blown oil could be mixed or blended only to a slight extent with American petroleum; it mixed, however, a little more freely with Scotch petroleum, and comparatively easily with Russian petroleum. Another peculiarity, probably due to the oxidized condition of the fatty acids, was that the sulphuric acid reaction increased out of proportion to the iodine value, whereas in the case of most oils a certain ratio existed between these two factors. The bromine thermal rise also did not bear a normal relationship to the iodine value; there was generally a low Hehner value and a low flash-point. The flash-point might be 100° lower than that of the original oil.

Mr. ALLEN said that although the author had stated that at present the exact bearing of all these figures could not be definitely laid down, nevertheless, the accumulation of data of this kind was extremely valuable, and would probably be followed by the removal of the difficulties which at present existed, and by a consequent better understanding of the chemistry of blown oils. These oils were of an extremely complex nature, and any method by which a proximate analysis of their constituents could be made was of great value.

Mr. HORATIO BALLANTYNE said that he was pleased to see that in their general aspect these figures accorded with the results obtained by Mr. R. T. Thomson and himself some ten years previously. One most interesting point was the very high percentage of soluble fatty acids which blown oils contained. In testing for the presence of blown oil in castor oils of doubtful purity, the percentage of soluble fatty acids—determined just as in the case of butter analysis—was probably the very best criterion by which to judge.

Dr. LEWKOWITSCH said that the miscibility of blown oils with petroleum distillates showed that, after all, in some sense the surmise was correct that these oxidized acids were hydroxy acids, because in that respect these oils approached castor oil, and, in fact, were known, when first brought into the market, as "soluble castor oil." In regard to solubility, they evidently took an intermediate position between castor oil and the other fatty oils. That Russian petroleum behaved somewhat differently from American petroleum could only be due to a difference in composition. Whilst the American petroleum distillates consisted chiefly of hydrocarbons of the paraffin series, the hydrocarbons of Russian petroleum largely belonged to the naphthene series, and must therefore exhibit a different solubility. He was afraid that he had not a very favourable opinion of the usefulness of the Maumené test and the bromine thermal test. It seemed to him much better to determine the iodine value itself. The low flash-point of blown oil was due to the considerable amount of volatile acids. Of course, the acetyl value, which might be indicative of the presence of castor oil, would break down in these cases. No doubt the determination of the percentage of volatile acids was a very good test indeed. It was somewhat puzzling to find such a large amount of volatile or soluble fatty acids in a product which from its treatment would hardly be expected to contain any volatile fatty acids at all.

THE DETECTION OF ARTIFICIAL COLOURING MATTERS IN FRESH AND SOUR MILK.

By M. Wynter Blyth, B.A., B.Sc., F.I.C.

(*Read at the Meeting, March* 5, 1902.)

In view of the recommendations* of the Departmental Committee on Preservatives and Colouring Matters in Food, 1901, we may expect the addition of colouring matters to milk to be at some time or other an offence under the Sale of Food and Drugs Act. On this account I think a few observations and experiments recently made by myself may be of some value to analysts.

The colouring matters which have been used from time to time in milk are numerous, and include annatto, turmeric, saffron, carotin, caramel and coal-tar dyes, but practically at the present time in this country the only colouring matters used to any extent are annatto and coal-tar dyes. That these latter are being used more and more both in milk and other articles of food in place of the natural colouring matters is a matter of common knowledge, and personally I found during the year 1901 no less than 19 per cent. of the retail samples of milk submitted to me for analysis under the Sale of Food and Drugs Act to be coloured with coal-tar dyes.

The coal-tar dye used at present is, as far as my own experience goes, methyl orange (salts of dimethylamido-azo-benzene-sulphonic acid), or dyes very closely allied to this body. Should, however, the addition of colouring matters to milk be prohibited, we may expect the adulterator, us usual, to exercise his ingenuity in order if possible to defeat the analyst, and we shall certainly find many other dyes used. For instance, salts of the aniline base—amido-azo-benzene (aniline yellow), or its dimethyl derivative (butter yellow), salts of amido-azo-benzene-sulphonic acid (acid yellow), salts of diphenyl-amine-azo-benzene-sulphonic acid (orange IV.), all impart a more or less suitable colour to milk; the other sulphonated azo dyes are not in a pure state so suitable for colouring purposes. Salts of dinitro-alpha-naphthol (Martius yellow), salts of dinitro-ortho and para-cresol (victoria yellow), and certain basic dyes, such as phosphine, might also be used, especially the latter; and if we take into consideration mixtures of various colours, the number of possible dyes becomes very large.

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* Recommendation B: "That the use of any preservative or colouring matter whatever in milk offered for sale in the United Kingdom be constituted an offence under the Sale of Food and Drugs Act."

So, although the colouring matters used at present are limited in number, and for the most part easy of detection in fresh milk, I have, in view of probable contingencies, sought for some general method which shall cover as far as possible these future developments. In this country the detection of colouring matters in milk has not received any special attention. H. Droop Richmond ("Dairy Chemistry," p. 139, 1899) recommends the precipitation of the colouring matter and casein with acetic acid, and subsequent extraction of the colouring matter with alcohol. In America A. E. Leach (*Journ. Amer. Chem. Soc.*, 1900, 207) has studied the subject, and devised an excellent general method for the detection of a few colouring matters in fresh milk.

The detection of artificial colouring matters in sour or decomposed milk has, as far as I know, not been previously studied.

The usual test for coal-tar dyes in milk is the addition of hydrochloric acid to the milk itself or to the separated curd, when a pink colour is obtained in the presence of some coal-tar dyes. This test may fail for two reasons: (1) The milk may not be quite fresh;* (2) a coal-tar dye may be present which does not strike a pink colour with hydrochloric acid.

THE ACTION OF DECOMPOSING MILK ON COLOURING MATTERS.

A. *Coal-Tar Colours.*—If a small quantity of an easily-seen dye, such as methylene blue, be added to milk, and if the milk be incubated at body temperature for a few hours, or allowed to stand at ordinary temperature for some days, the blue colour will disappear, leaving the milk quite white. If the sample be now vigorously shaken with air the blue colour will slowly return. If, on the other hand, the milk be made strongly yellow with such a dye as methyl orange, and incubated in the same manner, the yellow colour disappears, and cannot be restored by shaking with air, nor can any pink colour be obtained on the addition of hydrochloric acid. This change takes place with small quantities of dyes very quickly and at very low temperature. Commercial samples of milk dyed with methyl orange may give a pink colour even on the fourth day in cold weather, but in summer they will, as a rule, give no reaction after standing twenty-four hours in the warm laboratory. *The action of decomposing milk on coal-tar dyes is exactly the same as is the action of zinc dust and hydrochloric acid.* This may be proved by the fact that the same decomposition or reduction products may be extracted from a dye incubated in milk as may be extracted from the dye when reduced by zinc and hydrochloric acid, and those dyes which are reduced to colourless bodies by zinc and hydrochloric acid, and which are again restored to the original dye by oxidation, are also reduced to colourless bodies by incubation in milk and again restored to the original dye by shaking with air. For example, methyl orange breaks up under the action of reducing agents into dimethyl-para-diamido-benzene and sulphanilic acid. Milk treated with methyl orange and incubated, if rendered alkaline and treated with ether, will yield up to the ether para-diamido-benzene. On evaporating the ether a rose-coloured residue will be observed, especially at the edges of the separated fat. This may be identified as para-diamido-benzene by shaking with 10 c.c. of warm distilled water, separating the fat, and adding to the aqueous solution (1) 2 drops of normal sulphuric acid, (2) 2 drops of saturated sulphuretted hydrogen solution, (3) 2 drops of very dilute ferric chloride solution, and warming, when methylene blue will be formed. By operating as above, as little as 0·00002 gramme of para-dimaido-benzene may be detected in 10 c.c. of water, if at the same time a control-tube with the reagents and distilled water is treated in the same manner. This methylene blue reaction is easily obtained with the residue obtained from sour milk when the amount of methyl orange added in the first place is large; but owing to the ease with which para-diamido-benzene oxidizes during the manipulations necessary to isolate it, it is very difficult to obtain with the residue from commercial milks which contain between 1 part of colouring matter in from 100,000 to 300,000 parts of milk. The rose-coloured residue is, however, very characteristic, and may easily be observed, even without separating the fat, in the ether residue from 50 c.c. of commercial milk containing methyl orange.

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* To this reason, I think, must be attributed the fact that no coal-tar dye was found in any of the 296 samples examined in the Government Laboratory as recorded in the report of the Departmental Committee on Preservatives, etc., 1901. The Committee evidently recognised the difficulty of examining decomposing foods, for they state "that the characteristic reactions for the colouring agents are frequently interfered with or obscured by the organic matter of the food itself, especially after decomposition has commenced."

In the same manner orange IV. yields a residue of impure para-amido-diphenylamine, which with a drop of ferric chloride gives an intense blue-green colour changed to scarlet by strong sulphuric acid. Other dyes yield residues which are either coloured or become so on standing for a little time exposed to the air. A few examples of the nature of the decomposition and reduction products of different dyes are given in the table at the end of this paper, but the important point is, not the absolute recognition of the decomposition product of any dye, but the fact that milk which has never contained a coal-tar dye does not become coloured on shaking with air, and yields from the ether extract no coloured residue or any product which gives any colour reactions with such reagents as ferric chloride or strong mineral acids.

B. *Other Colouring Matters.*—Annatto, turmeric, saffron, carotin, and caramel are not reduced to colourless bodies by decomposing milk (except perhaps after a very long time).

The General Examination of Milk for Colouring Matter.

The colour of the milk should be carefully noted, as some dyes, such as aniline yellow and acid yellow, impart a faint pink colour to the curd of sour milk; butter yellow, on the other hand, rises with the fat, which it at first colours yellow, but afterwards becomes colourless; annatto imparts a characteristic colour to the curd, and, like saffron and turmeric, does not become colourless in decomposed milk;

caramel colours both the curd and the whey. If it be desired to make a comparison between the milk when fresh and after partial decomposition, two portions should be incubated, one containing sufficient formalin to prevent any decomposition.

The following general method will be found most convenient: Take 50 c.c. or more of the milk (as much as can be spared if reduction products are specially looked for) and render it just alkaline to delicate litmus paper, evaporate to a paste, and thoroughly extract the fat with ether. Although turmeric, annatto, and such dyes as aniline yellow are all somewhat soluble in ether from an alkaline solution, yet in the presence of casein such small quantities are dissolved that they may be disregarded. (Phosphine is much more soluble in ether, and should be looked for both in the ether and alcohol extract.) Evaporate the ethereal solution to dryness, shake up the fat with a small quantity of hot distilled water in a small separating funnel, separate the water from the fat, and evaporate to dryness on the water-bath in a small flat porcelain dish; carefully note the colour of the residue (see Table). Pure milk will give no coloured residue. Next, thoroughly exhaust the fat-free milk residue with absolute alcohol, pass the alcohol extract through a small filter, and evaporate to dryness in three or four small flat porcelain dishes; if unreduced artificial colouring matter be present the residue will be coloured orange, yellow, or brown. Wash one of the residues into a test-tube with a small quantity of water made acid with sulphuric acid, the sulphonated azo dyes will be at once indicated by the colour of the solution; shake the solution with ether, this will divide the colouring matters into two groups, as in the table: the natural colouring matters and the non-sulphonated acid coal-tar dyes—*i.e.*, the dyes precipitated by Weingärtner's tannin reagent—being soluble in ether, while the basic dyes and the sulphonated dyes are insoluble in ether. The ordinary tests may then be applied to the remaining portions of the dyes. For the sake of completeness I append to the table some confirmatory tests for the colouring matters most likely to be met with at present in commercial samples of milk:

A. ALCOHOL EXTRACT, COLOURED ORANGE, YELLOW, OR BROWN.

Take up a Portion of the Residue with Dilute Sulphuric Acid and shake with Ether.

THE ETHER DISSOLVES SOME OF THE COLOURING MATTER.		THE ETHER DOES NOT DISSOLVE THE COLOURING MATTER.	
Natural Colouring Matter.	Non-sulphonated Acid Coal-tar Colours, such as:	Basic Coal-tar Colours, such as:	Sulphonated Coal-tar Colours, such as:
Annatto	Aniline yellow	Phosphine	Acid yellow
Turmeric	Butter yellow	—	Methyl orange
Saffron	Victoria yellow	—	Orange IV.
Carotin	Martius yellow		

B. Water Extract from the Fat.

Note the Colour and apply various Reagents to the Dry Residue.

Colour of Residue.	Probable Original Colouring Matter.	Add a Drop to Ferric Chloride.	To the Ferric Chloride add Strong H_2SO_4.	Other Reactions.
Brown	Acid yellow	Dark green	Yellow, green on dilution	
Brown	Butter yellow	Dark blue-green	Yellow, green on dilution	To a slightly acid solution of the colouring matter add a few drops of H_2S solution, then ferric chloride, heat—magenta colour.
Brown	Aniline yellow	Yellow	Yellow	Strong H_2SO_4, or HCl, gives a delicate violet
Yellow	Martius yellow	Red	Yellow	Strong HCl gives a beautiful rose red colour
Yellow	Victoria yellow	Red	Yellow	Same as Martius yellow
Rose red	Methyl orange	Fugative scarlet	Yellow	Treat as with butter yellow, the solution becomes a beautiful blue.
Brown-red	Orange IV.	Green	Scarlet, green on dilution	Treat as butter yellow, the solution becomes dirty violet
Yellow	Unreduced dyes, soluble in ether from alkaline solution			

Confirmatory Tests for the more commonly used Colouring Matters.

Annatto.—(1) Take up the yellow alcohol residue with weak sodium hydroxide solution, dry a few drops on filter-paper. An orange spot changed to pink by stannous chloride solution indicates annatto. (2) Dissolve the yellow alcohol residue in a little water, add a little alcohol and a few drops of ammonia. Introduce a bundle of white cotton fibres and evaporate the liquid nearly to dryness on the water-bath; wash the fibre, which will be yellow, and then immerse it in citric acid solution, when it will become pink. This excellent test is due to A. Leys, who, however, isolates his annatto in a different manner. (3) A convenient method for dealing with a large number of samples is that proposed by Leffmann and Beam ("Select Methods of Food Analysis," p. 217, 1901). The milk is rendered alkaline by acid sodium carbonate, and a slip of filter-paper allowed to soak in it all night. Annatto causes a reddish-yellow stain on the paper.

Turmeric.—The usual test with boric acid is sufficient.

Caramel.—This is one of the most difficult of all colouring matters to detect in small quantities in milk. Whether it is used in this country is doubtful; it is, however, used in America to some extent (A. E. Leach, Thirtieth and Thirty-first Annual Reports, State Board of Health of Massachusetts). The following modification of the test proposed by A. E. Leach (*Journ. Amer. Chem. Soc.*, xx., 207) I have found to give the best results. It is useless to use any evaporation process, as even with great care some caramel may be formed from the milk-sugar. Obtain a milk known to be free from caramel as a control. Take 50 c.c. of the suspected milk and the same quantity of the control, coagulate each by the addition of acetic acid, strain off the whey from the curd by means of pieces of fine muslin; carefully compare the colours of the whey from both samples. Place the curds in two white porcelain basins and just cover them with strong hydrochloric acid. Compare the colours after they have stood several hours; caramel will be indicated by a brownish-violet colour, but the pure curd will also develop a similar colour after standing for a long time, so that great care must be exercised in coming to a conclusion. The use of the control milk greatly increases the delicacy of the test.

Sulphonated Azo Dyes.—These all strike characteristic colours with strong acids; their presence may be confirmed by allowing the milk to go sour and extracting the decomposition products, or, better, by reducing the extracted dye by zinc and hydrochloric acid, making alkaline with ammonia, extracting the ammoniacal solution with ether, and examining the ether extract.

NOTE.—Since reading the foregoing paper I have fully examined a number of milks for colouring matters. The following results were obtained during the month of March:

	Total Examined.	COLOURED.			Not Coloured.
		Coal-tar only.	Annatto only.	Annatto and Coal-tar.	
A. Retail town samples	50	9	14	6	21
B. Wholesale farmers' samples	29	None	None	None	29

These samples being few in number and representing only one month, are, nevertheless, interesting, in that they show the enormous difference between the town and farmers' samples. These observations are being extended so as to cover the whole year.

DISCUSSION.

Dr. RIDEAL thought that probably the reducing action to which the author had referred was due to micro-organisms. Dr. Pakes had pointed out about two years previously that organisms of the *coli* group produced hydrogen, and, if the reducing action was attributable to organisms of the *coli* group, the occurrence of the bleaching effect also indicated the presence of an organism which ought not to be present in a milk supply. In regard to the possible effect on health of the coal-tar colours mentioned, it seemed reasonable to look upon them as certainly less injurious than some preservatives were. They were present in very small quantities only, and even phosphine, which many people believed to be very poisonous, had been shown, in some experiments conducted by Professor Ray Lankester and himself, to be capable of being added to the food of small animals in considerable quantities and for considerable periods of time without producing any injurious effect.

Mr. Richmond said that the original object of adding colouring matters to milk was to preserve a continuity of colour between the milk produced from grass in the spring and summer and that produced from artificial food in the winter. Unfortunately, however, the practice had developed of preserving, not only continuity of colour in milk all the year round, but continuity between milk and skim milk. Certainly in a large number of commercial milks the colouring was completely overdone, and, to one accustomed to the appearance of milk without added colouring matter, a true effect was not obtained. A very simple test for added colouring matter was to let the milk stand. In the case of genuine high-coloured milk the bulk of the colour rose with the cream and the skim milk remained white, whereas with artificial colouring matter the bulk of the colouring matter would remain in the skim milk, unless the dye happened, as might occasionally be the case, to be one that was practically insoluble in water—such, for instance, as the dye of which methyl orange was the sulphonated derivative.

Dr. J. M. H. Munro inquired whether the author had tried the experiment of keeping a sample of sterilized milk at the incubating temperature, and if so, whether decolorization had been observed to take place, and whether decolorization took place after the addition of formalin. He would also like to hear whether the author could give any information as to the proportion which the milk samples coloured with coal-tar dyes bore to those coloured with annatto. It would naturally be imagined that annatto would be more largely used in the country, and coal-tar colours in and around London. He would like to hear what the author considered to be the easiest test for annatto. The simple test mentioned by Mr. Richmond was, of course, only applicable to whole milk, and not to separated milk.

Dr. Schidrowitz said that caramel, although not an injurious substance, sometimes had to be tested for—in spirits, for instance, when it was desired to ascertain whether a spirit was cask-coloured or coloured with caramel. The method generally recommended was that of Amthor, but his own experience with that test was by no means satisfactory. In the case of spirits that certainly were not coloured with caramel, he had obtained the precipitate with paraldehyde which Amthor described; while, on the other hand, a plain spirit, which he had coloured with caramel made by himself, yielded no precipitate whatever. There existed also an idea, which, however, was fallacious, that while naturally coloured spirit was decolorized on the addition of lead acetate or albumin, caramel-coloured spirit was not decolorized. In neither case was the decolorization perfect, although it was somewhat more marked in the case of the cask-coloured spirit.

Mr. Wynter Blyth said that he believed he was correct in stating that a pure culture of the typhoid organism in litmus milk did not decolorize the litmus, so that it would appear scarcely possible, from the fact that no decolorization occurred, to draw a safe conclusion that objectionable organisms were absent. He had found that if a poisonous colour, such as Martius's yellow, were added to milk, the poisonous colour acted as a preservative—an interesting fact which might possibly afford a means of detecting poisonous colouring matters. It would be interesting to see if phosphine acted as a preservative in that way. He was somewhat doubtful whether the simple test mentioned by Mr. Richmond would apply to small quantities of methyl orange. His experience was that methyl orange decomposed very quickly, and probably, on a warm day, by the time the cream had risen properly there would be very little methyl orange left. He had found that butter-yellow rose and coloured the cream, but he believed that this colour was not used very largely, owing to its insolubility in water. He did not think that sterilized milk had any action on artificial colouring matters. He had kept litmus—which decolorized very rapidly in decomposing milk—for several months in sterilized milk without any decolorization; and the same would be the case, of course, if formalin had been previously added to the milk. He thought that probably the reducing action was due to the evolution of hydrogen by micro-organisms, as Dr. Rideal had suggested. The effect was just the same as that of treatment with zinc and hydrochloric acid. It did not occur in the presence of formalin. He could not give any precise information as to the proportion of milk samples containing annatto (see note), but he thought it possible that annatto was used to a considerable extent by country milkmen. Out of two hundred samples taken at railway-stations, as the milk arrived from the farmers, only one was found to contain coal-tar colouring matter. The 19 per cent. referred to represented town samples only. The easiest test for annatto was the one proposed by Leffmann and Beam, and consisted in soaking a piece of filter paper in the milk made alkaline with sodium carbonate, when, if annatto was present, a brown colour was obtained on the filter paper after twenty-four hours.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

The Detection of Spent Tea-leaves by the Sublimation Test. A. Nestler. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1902, v., 245-247.)—For the detection of extracted tea-leaves the author has found the sublimation test (see ANALYST, A. Wynter Blyth, ii., 39) to be very useful. He has also met with a sample of tea containing spent leaves which had been re-rolled and stiffened by means of starch paste. As regards the delicacy of the test, it was found that a small piece of tea-leaf, weighing 0·0011 gramme, gave distinct theine crystals after ten minutes' heating. W. P. S.

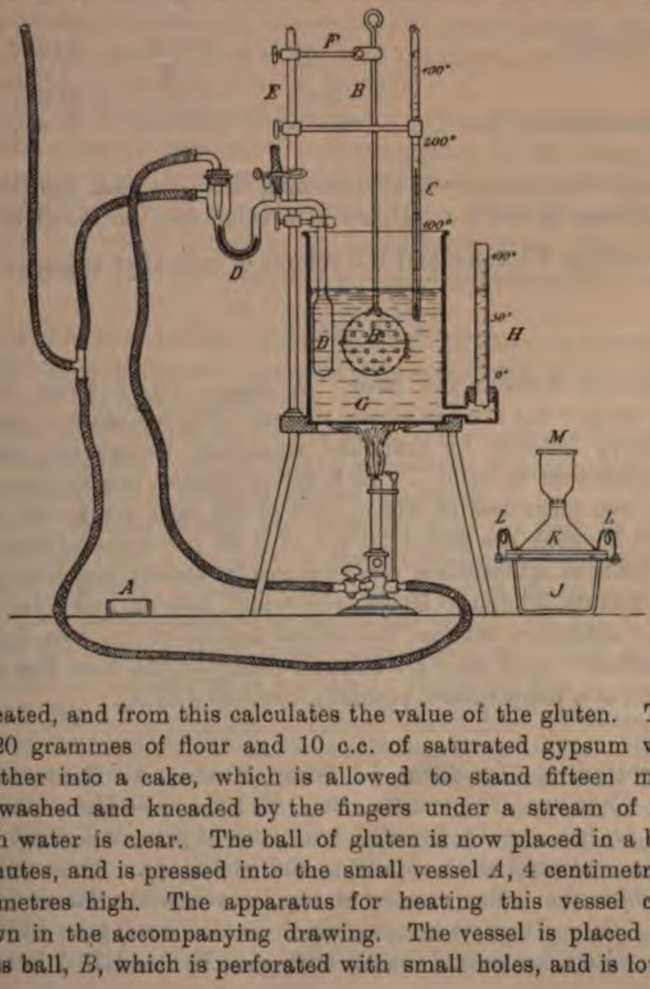
The Analysis of Meat Extracts and Vegetable Extracts. K. Micko. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1902, v., 193-210.)—In this paper are given analyses of various meat and vegetable extracts. The methods employed are those usually used for the separation of the different constituents of these articles. The author also describes methods for the estimation of the creatine and creatinine, and of the xanthine bases in the extracts.

Estimation of Creatine.—Five to 20 grammes of the extract are dissolved in a mixture of 100 c.c. of water and 10 c.c. of dilute sulphuric acid (1 : 3) and boiled under a reflux condenser for three hours. After neutralizing the liquid with sodium hydroxide solution, the xanthine bases are precipitated with acid sodium sulphite and copper sulphate, and filtered off. The filtrate is treated with sulphuretted hydrogen; after the addition of a little more dilute sulphuric acid the copper sulphide is filtered off, and the filtrate evaporated to a syrupy consistency. The residue is washed into a flask with boiling alcohol, and heated for some time under a reflux condenser until the creatinine has passed into solution. The alcoholic solution is then neutralized with alcoholic soda solution, and made just acid with a drop of dilute sulphuric acid. After standing some hours in the cold, the clear liquid is poured off and the residue once more extracted with boiling alcohol. The alcoholic liquids are evaporated, and the residue is again boiled out with alcohol, cooled and filtered. The solution thus obtained is evaporated down to about 300 c.c., cooled, and made feebly alkaline with alcoholic soda solution. The small precipitate which forms is filtered off, the filtrate is gently warmed on the water-bath, and 0·5 c.c. of a concentrated alcoholic solution of zinc chloride is added. The solution is allowed to cool, and the small amount of creatinine-zinc chloride which forms is filtered off. On reducing the filtrate to 30 to 50 c.c. by evaporation, the remainder of the creatinine-zinc chloride is deposited. The nitrogen in the precipitates is then estimated by Kjeldahl's process. Liebig's extract was found to contain approximately 6 per cent. of creatine by this method.

Estimation of Nitrogen occurring as Xanthine Bases.—Ten grammes of the extract are dissolved in 100 to 200 c.c. of water, and heated to boiling. Thirty to 40 c.c. each of saturated acid sodium sulphite solution and a 13 per cent. solution of copper sulphate are added, the solution is boiled for one minute, and allowed to stand overnight. The precipitate is then filtered off, washed with previously boiled cold water, and is placed, together with the filter-paper, in a flask, in which it is boiled, after the addition of a little water acidified with hydrochloric acid. When the paper has broken up, the copper is precipitated as sulphide, and removed by filtration. The cold filtrate is precipitated with silver nitrate solution, after first adding ammonia in sufficient quantity to dissolve the silver chloride which forms, and allowed to stand overnight. The precipitate is now filtered off, washed with dilute ammonia, then with water, and brought into a flask, where, after acidifying with hydrochloric acid, the silver is precipitated as sulphide. The filtrate from the silver sulphide is evaporated down and reprecipitated with ammonia and silver nitrate. This precipitate is washed with very dilute ammonia, then completely with water, and finally with alcohol. The nitrogen it contains is estimated by Kjeldahl's method.

The filtrate from the copper acid sulphite precipitate still contains some xanthine bases. To estimate these the copper is removed as sulphide; the filtrate from the copper sulphide is evaporated, precipitated with ammonia, and the small precipitate of phosphates which forms on standing is filtered off. The filtrate is then treated with ammonia and silver nitrate, as above. The amount of nitrogen found is added to the first quantity. Liebig's extract was found to contain 0·648 per cent. (average) of nitrogen as xanthine bases, "Sitogen" (yeast extract) 0·637 per cent. The author has not yet determined whether the nitrogen in the silver precipitate exists solely as xanthine bases, nor whether the latter are identical in meat and vegetable extracts. W. P. S.

Apparatus and Method for Determining the Quality of Gluten. Liebermann. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1901, iv., 1009-1016.)—The quantity of gluten in wheat does not measure its suitability for baking purposes. A wheat poor in gluten may yield better bread than does a wheat containing a larger quantity. The author, therefore, determines the volume to which the gluten will swell when heated, and from this calculates the value of the gluten. The method is as follows: 20 grammes of flour and 10 c.c. of saturated gypsum water are well kneaded together into a cake, which is allowed to stand fifteen minutes. The cake is then washed and kneaded by the fingers under a stream of gypsum water until the wash water is clear. The ball of gluten is now placed in a basin of water for fifteen minutes, and is pressed into the small vessel *A*, 4 centimetres in diameter and 1·5 centimetres high. The apparatus for heating this vessel containing the gluten is shown in the accompanying drawing. The vessel is placed in the hinged spherical brass ball, *B*, which is perforated with small holes, and is lowered into the



oil-bath, *G*, which has been previously heated to a temperature of 170° C. As soon as all air and steam have been driven off the level of the oil is read on the scale, *H*. The gluten is heated for fifteen minutes, being kept at a constant temperature by the thermostat, *D*, the level of the oil is again taken, and the sample is then withdrawn from the bath, allowing it to drain for a short time into the bath. Knowing the radius of the bath, the volume of the cooked gluten may be calculated. A simpler means of determining its volume is to measure the amount of alcohol the ball of gluten will displace in the apparatus *J*. Good gluten takes up an almost spherical shape on heating as above, whilst bad samples are not so regular in shape, and have a much smaller volume. The following examples will show the working of the method. It should be mentioned that, when the flour yields more than 40 per cent. of moist gluten, 10 grammes of the flour must be taken instead of 20 grammes.

	A.	B.	C.	D.
Quantity of flour taken	20 grs.	20 grs.	20 grs.	20 grs.
Nitrogen	2·10%	2·22%	1·61%	1·47%
Moist gluten	31·6%	35·2%	24·6%	22·3%
Volume of heated gluten	135 c.c.	120 c.c.	76 c.c.	32 c.c.

An arbitrary valuation is made by assuming that in a good flour the gluten cake expands to a volume of 348 c.c. Hence this value may be found by the formula $V = \frac{K \times 100}{348}$, in which V = value and K = volume of cake after expansion.

W. P. S.

Recognition of Pasteurized Beer. **A. Bau.** (*Wochenschr. Brauerei*, 1902, xix., 44.)—The author has already shown that all beers contain invertase, an enzyme which is rendered inactive by heat, and weakened, though not destroyed, by a temperature of from 50° to 57·5° C. In order to employ this enzyme as a test for pasteurization, two quantities of 20 c.c. each of the beer are taken; one is boiled, the other not. To each are added 20 c.c. of a 20 per cent. solution of sugar, the flasks are set aside at atmospheric temperatures for twenty-four hours, treated with 0·5 c.c. of lead acetate, diluted to 50 c.c. with water, filtered, and polarized. If there is a noteworthy difference between the optical activity of the two liquids, the beer was not pasteurized; but if they are alike, or nearly so, the beer had certainly been pasteurized, and at a temperature, probably, exceeding 57° C. F. H. L.

The Detection of Horseflesh in other Meats. **A. Hasterlik.** (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1902, v., 156-158.)—The author records two cases in which he has recently detected horseflesh in preserved meat (corned beef), the seller being convicted after admitting the admixture. The method employed was that in which the iodine number of the intra-muscular fat is determined (see ANALYST, xxii., 104). The first sample (called pickled beef) gave iodine numbers of 72·7 and 72·3, whilst the second (boiled beef) gave 78·9 and 78·5 in two determinations. A sample of genuine pickled beef gave iodine numbers of 56·0 and 55·4. Two other boiled beefs—one lean and the other fat—gave 57·7 and 43·6 respectively.

A. Schuster has communicated to the author the iodine numbers of the intramuscular fat of various samples of beef—roast beef, fillet, etc. The determinations—twenty-nine in all—gave results from 41·9 to 54·0, the average being 47·5. It was found that pickled beef gave a similar ring colouration with iodine (test for glycogen) as did pickled horse-flesh. W. P. S.

The Application of Serum Diagnosis to the Examination of Food. G. v. Rigler. (*Oesterr. Chem. Zeit.*, 1902, v., 97-100.)—In 1898 Bordet discovered that on injecting cows' milk into rabbits the serum from the animals thus treated gave a precipitate of casein when subsequently introduced into cows' milk. This discovery was supplemented by Wassermann, who, in 1900, found that it was possible in this way to distinguish between the milk of cows and of goats; and he suggested the use of these precipitating agents formed in the sera of animals (*precipitines*) as a general means of distinguishing between different proteids.

Subsequently, in conjunction with Fisch, he demonstrated that each species of animal possessed a specific milk capable of recognition by this test, the precipitate being obtained either with the whole milk or with that greatly diluted.

It was next shown by the experiments of Stern, Mertens, and others, that the serum of rabbits into which human serum, albuminous urine, pleuritic exudations, etc., had been injected was only capable of giving precipitates with those respective liquids.

Then Schütze applied the test to an extract of human muscle, and to vegetable extracts containing proteids, and suggested the application of the method to the examination of artificial food preparations.

Starting with this idea, the author has made experiments to determine whether the specific serum reaction could be used as a means of distinguishing between the cooked and uncooked flesh of different species of animals.

A 20 per cent. aqueous extract was prepared from the flesh of seven different kinds of animals, and from 5 to 10 c.c. of these extracts were injected beneath the skin of rabbits at intervals of three days. After a month the animals were killed, and the serum of the blood separated by centrifugal force.

In each case the supposed specific sera were mixed with clear, filtered aqueous extracts of the flesh of the respective animals in the proportion of 1 to 5, the tubes kept at a temperature of 37° C., and examined after one, two, and three hours.

It was found that the sera only gave a turbidity or precipitate with their corresponding extracts. Thus the serum from the rabbit treated with the extract of horseflesh only gave a reaction with that extract, and not with that of venison, beef, pork, etc. Similarly, the serum from a rabbit treated with an extract of rabbits' flesh reacted with extracts of rabbits' flesh, but not with extracts of the flesh of cats, hares, or other animals, and so on.

In the case of mixtures, the sera only reacted with the extracts of the flesh of the specific animals. Thus a rabbit treated with an extract from a mixture of the flesh of a hare, calf, deer, and pig, yielded a serum giving a reaction with the extracts of the flesh of each of those animals, but with none of the others. In like manner a serum obtained by injection of an extract of a sausage composed of veal and pork only gave a turbidity with extracts of the flesh of calves, or cows, and pigs. There was no distinction between the flesh of the cow and of the calf. The reactions were obtained as readily with extracts of boiled or roast flesh as with those from raw flesh.

A series of blank experiments with the normal sera of the rabbit, cow, and pig gave negative results with the extracts of all the different kinds of flesh.

The author also made experiments to determine whether the method would be applicable to the detection of different kinds of sugar in honey. It was found, however, that only honey gave any reaction.

E. Ruppin (*Zeit. für Untersuch. der Nahr. und Genussmittel*, viii., 356) has applied the above process successfully to the detection of horseflesh mixed with other flesh. He found that the flesh extract used for control purposes, if acid, gave a spontaneous precipitate under the conditions of the experiment. To avoid this, the author treats the extract with sodium hydroxide solution until it no longer reddens sensitive blue litmus paper, and also adds to the diluted extract (1 : 25) a single drop of 1 per cent. solution of anhydrous sodium carbonate. Flesh extracts so treated gave no precipitate with the serum of untreated rabbits after remaining for several hours in an incubation chamber kept at a temperature of 37° C. The author found that the preservatives potassium nitrate, sodium sulphite, salicylic acid, boric acid, or smoking the meat, were without influence on the reaction. A mixture of minced beef with 5 and 2 per cent. of horseflesh gave the reaction distinctly. The process was tried on various sausages: a reaction was invariably obtained with these when uncooked, slight cooking materially lessened it, and prolonged cooking entirely destroyed it.

C. A. M.

The Alleged Poisonous Properties of "Mandarin" and "Metanil Yellow." G. W. Chlopin. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1902, v., 241-245.)—The author finds that "metanil yellow" is harmless, even when given in daily doses of 2 to 3 grammes to dogs and 0·2 gramme to human beings. On the other hand, he does not agree with the statement of Frentzel that "mandarin" is innocuous in moderate quantities (see Analyst, 1902, xxvii., 92-93). Quantities of this colour, given in daily doses of 2 grammes to a dog, caused uneasiness, vomiting, and diarrhœa. The author himself took 0·2 gramme, and the symptoms were so alarming (dizziness, headache, etc.) that the substance had to be removed from the system by means of a purgative.

W. P. S.

Presence of Copper in Powdered Drugs and Chemicals. E. H. Gane. (*Journ. Soc. Chem. Ind.*, xxi., 224.)—Some consignments of powdered ammonium carbonate having developed a blue mottled appearance in consequence of the presence of this impurity, it became necessary to ascertain definitely its origin. It was finally traced to the copper wire or rivets used to fasten the belts that drove the mill. As the belt wore down small fragments of copper were thrown off and fell into the hopper. The leather belt having been replaced by one of rubber, the trouble ceased. A. M.

ORGANIC ANALYSIS.

Estimation of Potato Starch (Granulose). A. Kaiser. (*Chem. Zeit.*, 1902, xxvi., 180.)—The sample is gelatinized in water in the usual way, so as to form a solution of about 1 per cent. strength, and this must be newly made. Fifty c.c. of the liquid are mixed with 10 grammes of sodium acetate, warmed to 50° C., and then precipitated with 25 c.c. of a solution containing 5 grammes of iodine and 10 grammes of potassium iodide per litre, taking care that a small excess of iodine is present. When the precipitate has settled it is collected on a tared paper and washed with a solution of 3 grammes of sodium acetate in 100 c.c. of water. It is next rinsed by means of alcohol into a porcelain basin, and gently warmed with about 5 c.c. of 5 per cent. alcoholic potash until the change of colour shows that the iodide of starch has decomposed. Sufficient excess of alcoholic acetic acid is then added, and after standing some time (the starch retains alkali with much persistence) it is brought on to the same paper as before, washed with hot spirit, absolute alcohol, and ether, and finally dried for four hours at 120° C. The starch so obtained should be weighed between watch-glasses because of its hygroscopicity; and its purity should be tested by its complete solubility in water, any insoluble cellulose, etc., being dried, weighed, and deducted. F. H. L.

A Method of Determining the Solubility of Alkaloids. R. A. Hatcher. (*Amer. Journ. Pharm.*, 1902, lxxiv., 134-137.)—This method is intended for determining the solubility of alkaloids which do not readily dissolve in water: 0·1 gramme of the alkaloid is rubbed with water so as to form a smooth paste, and then with more water until a smooth mixture of about 9 c.c. is obtained. To this is added from a burette sufficient normal sulphuric acid to just convert the alkaloid into sulphate, and then sufficient water to make 10 c.c. in all.

Portions of 1 c.c. each of this solution are diluted with different proportions of water, and a slight excess of normal sodium hydroxide solution added to each, the solubility being estimated from the relative amounts of precipitate.

Two fresh portions of 1 c.c. are then diluted approximately to the neutralization point, one being taken slightly above and the other slightly below that found in the first determination, and the sodium hydroxide added as before. Finally, a third determination based on the second results will give the solubility by taking the mean between the highest result giving a precipitate and the lowest showing none.

Thus, in the case of cinchonine, a slight precipitate was observed on adding a slight excess of sodium hydroxide to a solution of the sulphate containing 1 part in 22,000, but none in that containing 1 in 24,000, and the solubility was therefore taken as 1 in 23,000.

Applying the test to morphine, the solubility of which is known to be 1 in 4,350, the author found that on diluting the portions of 1 c.c., and then adding a very slight excess of alkali, a slight turbidity was given with the liquid diluted to 43 c.c., but none with that diluted to 44 c.c. The solubility was therefore taken as the mean of these results, or 1 part in 4,350.

C. A. M.

Detection and Estimation of Lactic Acid in the Gastric Juice. A. C. Vournasos. (*Zeits. f. angew. Chem.*, xv., 172.)—For the detection of lactic acid the author makes use of a reagent containing iodine and methylamine. Half a gramme of potassium iodide is dissolved in 50 c.c. of water, 1 gramme of iodine is added, the mixture is well shaken, then filtered, and 5 grammes of methylamine are added. Of the gastric fluid under examination, 5 c.c. are filtered and, if viscous, diluted. It is made strongly alkaline by the addition of a little 10 per cent. potash solution, then 1 c.c. of the above reagent is added. The lactic acid is converted into iodoform, which, with the methylamine, gives isocyanide:

$$2C_3H_6O_3 + 5K_2O + 12I = 4HCO_2K + 6KI + 2CHI_3 + 3H_2O$$
$$CHI_3 + 3KHO + CH_3NH_2 = 3KI + CH_3NC + 3H_2O.$$

The penetrating smell of the isocyanide enables one to detect 0·005 per cent. of lactic acid.— If less than that quantity be present, a larger amount of the sample should be taken and extracted with ether, the ether extract evaporated, and the residue tested as above. If volatile fatty acids are present, the gastric juice should be evaporated at 100° C. to a third of its volume.

For the gravimetric estimation of the lactic acid take 30 c.c. of the sample, and evaporate it to one-third. Then mix in a retort with 15 c.c. of aqueous caustic potash and 0·5 gramme of sublimed iodine. The mixture is distilled cautiously into a cooled receiver until $\frac{7}{10}$ have passed over. The distillate contains the whole of the iodoform from the lactic acid present. The iodoform may be collected on a tared filter, washed with water, and dried at 60° C. to constant weight.

The determination may also be made volumetrically. To the distillate obtained as above are added 50 c.c. of water and the same quantity of 10 per cent. alcoholic potash solution. The mixture is stirred till clear, then titrated with decinormal silver nitrate solution by an application of the precipitation method for iodides.

A. M.

The Detection of Indican in Urine. Bertault. (*Journ. Pharm. Chim.*, 1902, xv., 277-279.)—In testing urine for indican the blue coloration is sometimes masked by a violet colour, due to the presence of iodine.

In such cases the following modification is recommended: The urine is mixed with an equal volume of hydrochloric acid and shaken with a few c.c. of chloroform and a few drops of a dilute solution of sodium hypochlorite.

The acid layer is removed by means of a pipette, and the chloroform washed with water and then rendered just alkaline by the addition of a few drops of potassium hydroxide solution. The violet colour due to the iodine disappears, and the chloroform remains blue in the presence of indican, or becomes colourless in its absence.

Care must be taken not to add too large an excess of hypochlorite.

C. A. M.

INORGANIC ANALYSIS.

The Precipitation of Metals by Bismuth Oxide. J. Aloy. (*Bull. Soc. Chim.*, 1902, xxvii., 136, 137.)—Lebaigue (*Journ. de Pharm.*, xxxix., 51) stated that bismuth oxide, when boiled for about fifteen minutes with solutions of metallic salts, precipitated ferric, aluminium, and chromium in the form of hydroxides, but was without action upon ferrous, cobalt, nickel, copper, and lead salts. He recommended the reaction as a means of separating these classes of metals in analysis.

The author, however, finds that the reaction is not so simple as Lebaigue supposed, and that it is not applicable to analytical work. On boiling freshly-precipitated bismuth oxide for an hour with solutions of the different salts, he obtained the following results:

Ferric Salts.—The iron was only completely precipitated from very dilute solutions. The precipitate consisted of oxide.

Ferrous Salts.—Bismuth oxide precipitated the iron in the cold, and more rapidly on boiling, as a white amorphous precipitate, probably consisting of a mixture of basic salts.

Aluminium was only completely precipitated from very dilute solutions. *Manganese* yielded a precipitate of hydroxide, but the precipitation was only partial. *Copper* was also partially precipitated from concentrated solutions, yielding bluish-green basic salts in the case of the chloride, sulphate, and nitrate, and as black hydroxide from a solution of the acetate.

Cobalt, nickel, lead, zinc, and *cadmium* were also partially precipitated from solutions of their salts.

Anhydrous bismuth oxide behaved in the same way as the hydroxide, but the reactions took place more slowly.

C. A. M.

On the Influence of Hydrochloric Acid on the Precipitation of Cuprous Thiocyanate. R. G. van Name. (*Zeits. anorg. Chem.*, xxx., 122.)—Continuing his investigations on the precipitation of cuprous thiocyanate (*Zeits. anorg. Chem.*, xxvi., 230), the author found that the precipitation is practically complete, provided the amount of free hydrochloric acid left after the addition of the ammonium bisulphite and thiocyanate does not exceed 0·5 per cent. of the total volume of the solution, and also provided that the solution does not contain an excessive amount of ammonium salts, which exert a solvent action on the precipitate. The addition of a moderate excess of ammonium thiocyanate, however, is recommended if the above conditions cannot be satisfied, as cuprous thiocyanate is more insoluble in weak solutions of this salt than in pure water, solutions of other ammonium salts, or dilute hydrochloric acid.

A. G. L.

Use of Phosphoric Acid in Analysis of Iron Alloys. T. H. Byron. (*Journ. Soc. Chem. Ind.*, xxi., 214.)—Ferrosilicon, spiegeleisen, etc., dissolve readily in phosphoric acid, but the silica is left in such a gelatinous condition that filtration is impracticable. Phosphoric acid may, however, be used with advantage to determine chromium in ferro-chromium alloys and in chrome iron ores. The chromium is oxidized with permanganate and titrated with ferrous sulphate and bichromate. The results agree with those obtained by the older, more laborious method. A. M.

Estimation of Sodium Thiosulphate, Sulphite, and Sulphide.—Dupré and W. Korn. (*Zeits. f. angew. Chem.*, xv., 225.)—In order to estimate thiosulphate in the presence of sulphite the latter is decomposed, by boiling with about four equivalents of acetic acid and twelve of sodium acetate for one or two hours. Under these conditions thiosulphate is not decomposed and can therefore be titrated with iodine solution. The time of boiling may be reduced to about half an hour if the last traces of sulphite are removed by oxidation with potassium chlorate. About a gramme of chlorate is added, and the boiling is then discontinued.

If thiosulphate, sulphite, and sulphide all be present, they are first all determined by running into iodine solution acidified with hydrochloric acid. In a second portion the sulphide is precipitated by the addition of cadmium carbonate. The solution is filtered, and the filtrate is divided into two portions. In one portion thiosulphate + sulphite are determined, and in the other thiosulphate is estimated as above. From the three values thus obtained the individual constituents can be calculated by subtraction. A. M.

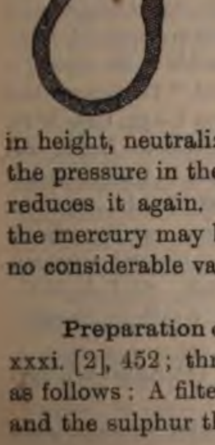
The Quantitative Separation of Hydrochloric and Hydrocyanic Acids. Theodore William Richards and Sidney Kent Singer. (*Amer. Chem. Journ.*, xxvii., 205.)—The authors find that hydrocyanic and hydrochloric acids can be quantitatively separated by boiling their aqueous solution for some time, preferably in a common retort, the neck of which is directed upwards at an angle of 45°, the evaporated water being replaced from time to time. The solutions should not be more than tenth-normal in regard to the hydrochloric acid. When little or no hydrochloric acid is present, as in the estimation of the chloride contained in potassium cyanide, two hours' boiling is sufficient to remove all the hydrocyanic acid; but when the solution contains a fair quantity of hydrochloric acid eight or nine hours' boiling is requisite. The difference in the behaviour of the two bodies on boiling is due to their very great difference in dissociation in aqueous solution; and the fact that in the presence of hydrogen chloride a longer boiling is necessary to expel all the hydrocyanic acid appears to be due to the formation of complexes between the chlorine ions and the undissociated hydrocyanic acid. A. G. L.

The Behaviour of Nitric and Nitrous Acids with Brucine and Sulphuric Acid. L. W. Winkler. (*Zeits. f. angew. Chem.*, xv., 170.)—In reply to Lunge's paper (*ibid.*, xv., 1), Winkler finds that the reaction of nitrous acid with brucine and sulphuric acid depends upon the proportion of sulphuric acid present. If equal volumes of concentrated acid and water be used deep red coloration results. With 2 volumes acid to 1 of water—the proportions recommended by Winkler for water analysis—a rose coloration is produced. With stronger acid the colour becomes less pronounced, and when 4 volumes sulphuric acid are present to 1 volume water—the proportions used by Lunge (*loc. cit.*)—there is no reaction in consequence of the formation of nitrosylsulphuric acid. The results obtained by Lunge are thus brought into agreement with those of Winkler.

On the other hand, nitrates give no reaction unless the acid be strong. With 2 volumes water to 1 volume sulphuric acid there is no coloration, but with 1 volume water to 2 volumes sulphuric acid a nitrate gives a coloration equal in intensity to that given by an equivalent amount of nitrite. By taking advantage of these peculiarities it is possible to test a water separately for nitrates and nitrites, using the same reagents, brucine and sulphuric acid. Lunge (*Zeits. f. angew. Chem.*, xv., 241) confirms Winkler's results and withdraws his previous remarks. A. M.

APPARATUS.

Pressure Regulator for Distillations under Diminished Pressure. W. Burstyn. (*Oesterr. Chem. Zeit.*, 1902, v., 55.)—This is an apparatus designed to keep the pressure inside a distillation-flask automatically constant within certain limits, so that variations in boiling-points, etc., may be avoided. A and B are charged with mercury, and S and D lead to the pump and distilling apparatus. Before beginning to exhaust, the stopper of A is removed, and the vessel is lowered till it is completely full of mercury; then, carefully excluding air, the stopper is put back. The pump is next set going, and when a sufficient vacuum has been attained (say, 20 millimetres) A is raised until the mercury in B touches the lower end of the tube D, thus producing a Torricellian vacuum in A. If, now, the pump becomes more powerful, so that the pressure falls to, say, 15 millimetres, the mercury takes the positions indicated by the dotted lines; but the pressure in the distillation apparatus remains unchanged, because the interposed mercury column, 5 millimetres in height, neutralizes the difference. Conversely, if by evolution of gas or otherwise, the pressure in the apparatus rises to more than 20 millimetres, the pump at once reduces it again. For more highly reduced pressures, such as 5 millimetres or less, the mercury may be replaced by petroleum oil, which is much lighter, and possesses no considerable vapour tension. F. H. L.



Preparation of a Fine-grained Filter-paper. E. Reale. (*Gazz. chim. Ital.*, 1901, xxxi. [2], 452; through *Chem. Zeit. Rep.*, 1902, 68.)—Such a paper may be prepared as follows: A filter is placed in a stoppered funnel, filled with ammonium sulphide, and the sulphur thrown down with 1 : 2 sulphuric acid. After standing five minutes the cock is opened, the liquid run off, and the paper washed thoroughly with water. Such a prepared filter-paper will be found useful for many purposes. F. H. L.

MILK-BLENDED BUTTERS.

KING'S BENCH DIVISION.

(Before the Lord Chief Justice of England, Mr. Justice Darling, and Mr. Justice Channell.)

PEARKS, GUNSTON, AND TEE (LIMITED) *v.* WARD—HENNEN *v.* SOUTHERN COUNTIES DAIRIES COMPANY (LIMITED).

From the " Times," April 24, 1902.

The first of these cases was an appeal from a conviction by the justices of Richmond, Surrey, under the Sale of Food and Drugs Act, 1875, Section 6. Three informations were preferred by the respondent against the appellants under this section, charging them with having sold to the prejudice of the purchaser butter which was not of the nature, substance, and quality demanded, the butter having had water added beyond the usual limit of 16 per cent. natural to butter. The magistrates convicted the appellants, and fined them £25 and costs. The appellants were a limited company under the Companies Act, and carried on business as provision merchants. The respondent, who was an agent of the Butter Association, caused one Annie White to purchase on his behalf at the appellants' shop in Richmond three packets of butter, which on analysis were found to contain respectively 24·7, 23·7, and 20·4 per cent. of water. It was admitted by Annie White that she understood Pearks' butter was moist, and that she could see it was moist, but that she asked for butter and expected to get butter. It was also admitted by respondent that he was not surprised on seeing the result of the analysis to be that the butter was adulterated with water, but that when the purchase was made he expected to get pure butter. It was proved that the excess of water was due to added milk. There was no evidence that at the time of sale the attention of the purchaser was drawn to the nature and composition of the article. The magistrates found as a fact that the purchaser did not know that the butter asked for contained such an excessive amount of moisture as was shown by the analysis.

Mr. Macmorran, K.C. (Mr. Joseph Ricardo with him), for the appellants, contended that, as the appellants were a limited company, they could not be convicted. Section 6 of the Act referred to " the person "; and though the Interpretation Act, 1889, Section 2 (1), enacted that the word " person " in a statute should include corporation, unless the contrary intention appeared, *mens rea* on the part of the person accused was necessary, and this was impossible in the case of a corporation. He referred to " Betts *v.* Armistead " (20 Q.B.D., 771) and " Pharmaceutical Society *v.* London and Provincial Supply Association " (5 Q.B.D., 310, and 5 App. Cas., 857). Further, there was no sale to the prejudice of the purchaser, as White knew that the butter was moist and the respondent was not surprised at the analysis. That amounted to this—that the respondent knew what he was buying. He cited " Mason *v.* Cowdary " (1900, 2 Q.B., 419).

Mr. Morton Smith, for the respondent, submitted that want of *mens rea* was no defence under this section. It was so held in " Betts *v.* Armistead." A master employing a servant was responsible under this Act for the acts of the servant—" Brown *v.* Foot " (61 L.J., M.C., 110)—unless the master proved that the servant was expressly prohibited—" Kearley *v.* Tonge " (60 L.J., M.C., 159).

Mr. Macmorran, in reply, cited " Abrath *v.* North-Eastern Railway " (11 App. Cas., 247) and " Hotchin *v.* Hindmarsh " (1891, 2 Q.B., 181).

The second of the cases was an appeal from a decision of justices acquitting the respondent company in proceedings under Section 6 of the Sale of Food and Drugs Act, 1875, on the ground that the section did not apply to corporate bodies.

Mr. RICKETTS argued the case on behalf of the appellant; the respondents were not represented.

The court gave judgment in the two cases together, dismissing the appeal in the first case, and allowing the appeal in the second.

The LORD CHIEF JUSTICE, in delivering judgment, said that in the first case the prosecution was under Section 6 of the Sale of Food and Drugs Act, 1875, for selling Pearks' butter, and it was contended that the information would not lie because the defendants were a limited company. There was another point—namely, that there had been no sale to the prejudice of the purchaser, because the woman who actually made the purchase knew that she would get moist butter. With regard to the latter point, it seemed to him that they had not to deal with the knowledge of the actual purchaser, except so far as the actual purchaser had been informed by the seller, because, of course, the person who bought on behalf of the inspector generally had knowledge, but the court had to consider the case of an ordinary purchaser. It was also impossible to hold that a statement by the woman that she knew that the butter was moist imputed knowledge of the extent of extra moisture in the article in question in the case. As to the question of the liability of a corporate body, which was the important question, it seemed to him very much the same question as that arising in civil proceedings as to the responsibility of the master for the acts of his servant. A corporation ought to be within the provision in question unless *mens rea* was an element of the offence. The language of Section 6 of the Sale of Food and Drugs Act was: "No person shall sell to the prejudice of the purchaser any article of food . . . which is not of the nature, substance, and quality of the article demanded by such purchaser under a penalty." Section 2 (1) of the Interpretation Act, 1889, provided that "in the construction of every enactment relating to an offence punishable on indictment or on summary conviction . . . 'person' shall, unless the contrary intention appears, include a body corporate." He could not see in Section 6 of the Act of 1875, or in any of the circumstances involved in what was contemplated by that section, any contrary intention whatever. It was clear that with reference to Sections 3 and 5 different considerations arose, but with reference to Section 6 he thought the point really covered in principle by the decisions. The protective object of the section and the expedients of the offence alike pointed to an intention to put a corporation in the position of an individual. He came to the conclusion that there was nothing within the meaning of the Interpretation Act disclosing a contrary intention. He thought, therefore, that the section applied to a corporation. The appeal in the first case must be dismissed. In the second case the appeal must be allowed, and the case remitted to be further dealt with.

Mr. JUSTICE DARLING and Mr. JUSTICE CHANNELL delivered judgments to the like effect.

HAYES *v.* RULE AND LAW.

This was an appeal by way of special case from a decision of the justices for the Redditch Division of Worcestershire dismissing an information laid by the appellant, a police inspector duly authorized to act under the Sale of Food and Drugs Acts, against the respondents under Section 6 of the Sale of Food and Drugs Act, 1875, charging the respondent Rule with selling as butter an article not of the nature, substance, and quality of the article demanded, and charging the respondent Law with aiding and abetting in the commission of the offence.

The facts stated in the case were to the following effect: On September 26, 1901, a Mrs. Dutfield, the wife of a police constable, acting under the direction of the appellant, went to a retail shop of Messrs. Pearks, Gunston, and Tee (Limited), in Redditch. She asked the shop-assistant Rule (the respondent) to supply her with half a pound of best fresh butter. It was supplied to her, and she paid 6d. for it. Shortly after this Mrs. Dutfield pointed to another lump on the counter, and said she would have half a pound of that butter. It was supplied and handed to her, and she paid 5½d. for it. At this point the appellant came into the shop, and the requirements of the Act were complied with. Each lot of butter when handed to Mrs. Dutfield was wrapped in a paper bearing the following statement: "This is choicest butter blended with pure English full-cream milk, whereby the percentage of water in the butter is increased to about 24 per cent." When the appellant came into the shop the respondent Law (the manager of the shop) pointed to a printed notice hung in the shop in these terms: "All butter sold at this establishment is Pearks' milk-blended butter, and is choicest butter blended with pure English full-cream milk, whereby the percentage of water in the butter is increased to about 24 per cent." This notice was hanging in such a position as to be facing and readily seen by any purchaser coming into the shop, though the appellant denied having seen the notice up to the time he began dividing the butter, but he admitted that, if he had so wished, he could have read the notice. There were two such notices in the shop. The appellant before purchasing had read of cases affecting Pearks' butter, and knew how the company were trading, but did not know anything about butter. The certificate of the analyst, in which he stated that in his opinion the sample of butter had been adulterated by the addition of water to the extent of 4·6 per cent., was put in. It was contended on behalf of the appellant that the wrapper and notice did not protect the respondents; that it had not been proved that the notice was seen by the purchaser prior to the completion of the purchase; that the recognised standard of moisture in butter was 16 per cent., whereas the analyst's certificate proved that the butter, the subject of the charge, contained 20·6 per cent.; that the onus lay upon the respondents to show how the excessive amount of moisture came to be in the butter, and that if they failed to satisfy the justices that it arose from causes beyond their control, or was not intentionally introduced by mixing milk with the butter, then the offence was complete, and no label they could frame would protect them against what was alleged on the appellant's behalf to be the fraudulent purpose of the respondents. It was contended on behalf of the respondents that the appellant had not made out a case; that the notice which had been settled for the respondents' employers by a learned King's counsel entirely protected the respondents; that there was not any legal standard as to the percentage of water in butter; that the prosecution had no case, the notice clearing the respondents from liability and disposing of any plea that the appellant might urge of fraudulent intent; that the appellant had admitted that before he went to the shop he knew what he went to purchase; that before sending Mrs. Dutfield to make a purchase the appellant admitted having read of previous cases and knew how the respondents' employers were trading, that they were selling milk-blended butter, and did not require notice to be put prominently before him. It was also contended that the notice protected the respondents from the charge that the goods were sold to the prejudice of the purchaser. The justices determined that as the appellant had by his agent asked to be supplied with butter, and had received an article which was not pure butter, although an article of commerce, the sale was a sale to the prejudice of the purchaser. But they held that the respondents were entitled to the protection afforded by Section 8 of the Act by reason of the printed notices exposed in the shop, and by reason that the butter sold to the appellant was wrapped in a printed notice disclosing the fact that the article was a mixture, and having regard to the admissions of the appellant that he knew how the respondents' principals were trading. The justices accordingly dismissed the information subject to the present case.

Mr. Amphlett, K.C., and Mr. Carmichael appeared for the appellant; and Mr. Asquith, K.C., Mr. Avory, K.C., and Mr. Frampton for the respondents.

The court yesterday dismissed the appeal.

The Lord Chief Justice said that in this case the court was asked to set aside an acquittal on a prosecution under Section 6 of the Sale of Food and Drugs Act, 1875. His Lordship wished the ground on which he thought the acquittal right to be distinctly understood, in order that the judgment of the court might not be supposed to involve larger issues than it did. The present case was, in his opinion, essentially different from that of "Pearks, Gunston, and Tee v.

Houghton" (18 the *Times* Law Reports, 362), which had been recently before the court, in that in the present case one had the purchaser asking for half a pound of the best fresh butter. The justices had found that what was supplied was not best fresh butter; so far, therefore, there appeared to be a sale to the prejudice of the purchaser. Under these circumstances, speaking for himself, he did not think it would have been sufficient for the respondents to rely on the notice hung in the shop without showing that it was called to the attention of the purchaser, because, as the purchaser asked for best fresh butter and nothing further was said, there would remain the question whether he saw it. But the justices had found that the respondents were protected by Section 8, because the article was sold with a notice disclosing the fact that it was a mixture. Unless that notice was so put round the butter that it would not be seen or was covered over with another paper, or something of that kind, the justices were quite right in holding it a good notice under Section 8. Mr. Amphlett had contended that there ought to be a specific finding that the label would have been seen by the ordinary purchaser. But unless that had been so, the justices would not have found that the article was wrapped in a notice "disclosing the fact that the article was a mixture." The other point raised was that there was no finding in the case negativing the suggestion that the admixture of the water was intended fraudulently to increase the bulk, weight, or measure of the article. But it was plain that if that point had been raised before the justices they must have intended to negative fraud. Therefore he thought that in this case the label was a good defence under Section 8.

Mr. JUSTICE DARLING and Mr. JUSTICE CHANNELL concurred.

KING'S BENCH DIVISION.

(Before the LORD CHIEF JUSTICE OF ENGLAND, Mr. JUSTICE DARLING, and Mr. JUSTICE CHANNELL.)

BAYLEY *v.* PEARKS, GUNSTON, AND TEE (LIMITED).

From the "Times," May 1, 1902.

THIS was an appeal by way of special case from a decision of three justices for the county of Southampton dismissing an information laid by the appellant against the respondent company under Section 8 of the Sale of Food and Drugs Act, 1899, charging them with selling to the appellant a quantity of margarine the fat of which contained more than 10 per cent. of butter fat. The substance in question was "Pearks' milk-blended butter," and was sold as such, with a label stating that it was "choistest butter blended with pure English full-cream milk, whereby the percentage of water in the butter is increased to about 24 per cent." The usual proceedings under the Sale of Food and Drugs Acts were gone through. The analyst's certificate which was put in stated that the substance contained 72·35 per cent. fat, 23·30 per cent. water, 2·55 per cent. lard, and 1·80 per cent. salt, and that the "butter" was adulterated with excess of water to the extent of 7·3 per cent. The analyst was called as a witness. He proved that the 72·35 was butter fat, and was the only fat present; that the texture of this butter differentiated it from ordinary butter; he was sure there had been a secondary manufacture, and that, in his opinion, it was a compound article; it was not adulterated with any other substance than water. On behalf of the appellant it was contended that this milk-blended butter was margarine, and must be dealt with under the Margarine Acts. It was not the substance usually known as butter, but was compounded of certain ingredients—namely, butter properly so called and milk unconverted into butter, and underwent a second process of manufacturing. It was made by mixing two kinds of butter together by means [of adding milk or cream. It was a mixed article and made in imitation of butter, and came within the definition of margarine in Section 3 of the Margarine Act, 1887. On behalf of the respondents it was contended that the article was butter, that it was admitted to be two butters blended together and could not be called an imitation of butter. That margarine was made from beef fat or some other like substance, and that if mixed with butter became margarine; but that it could not be contended that two butters mixed together became margarine or were anything else than butter. That the article came within the definition of butter in Section 3 of the Margarine Act, 1887. That the article was made exclusively from milk or cream only, and no other substance whatever was mixed with it. The justices were of opinion that the article in question was not margarine as defined by the Margarine Act, 1887, and they dismissed the information, subject to the present case.

Mr. Danckwerts, K.C. (with whom was Mr. Emmanuel), contended that the substance was "margarine" within the meaning of the Margarine Act, 1887, by reason of the definition of "margarine" in Section 3 of that Act. This definition was incorporated into the Act of 1899. The substance therefore came within the prohibition in Section 8 of that Act against selling margarine of which the fat contains more than 10 per cent. of butter fat.

Mr. Asquith, K.C. (with whom were Mr. Macmorran, K.C., Mr. Frampton, and Mr. Ricardo), contended that the substance was not margarine within Section 8 of the Act of 1899. It was not necessary to read into that section the definition in the Act of 1887 in all its amplitude. Further, the substance was not "margarine" within the definition in the Act of 1887. It was butter within the definition of butter in that Act. True, if a purchaser asked for butter simply, this substance would not be sold to him without explanation, because it was not the ordinary butter the purchaser must be taken to demand. But it did not follow it was not butter within the definition of the Act of 1887.

The court dismissed the appeal.

The Lord Chief Justice, in giving judgment, said that the information was for selling margarine the fat of which contained more than 10 per cent. of butter fat. The magistrates had dealt with the information by holding that the substance sold was not margarine. The force of Mr. Danckwerts' argument rested upon the following facts: When the Legislature enacted Section 8 of the Sale of Food and Drugs Act, 1889, it used the word "margarine." There was in the Margarine Act, 1887, a definition clause enacting that "'margarine' shall mean all substances, whether compounds or otherwise, prepared in imitation of butter, and whether mixed with butter or not, and no such substance shall be lawfully sold, except under the name of margarine, and under the conditions set forth in this Act." By Section 25 of the Act of 1899 expressions in that Act were to have the same meaning as in the Sale of Food and Drugs Acts and by Section 28 of that Act the Margarine Act, 1887, was included among these Acts. Mr. Danckwerts had contended that the expression "margarine" must include the substance now in question, because the definition from the Act of 1887 had to be applied, and, since it was prepared in imitation of butter, it could only be sold under the name of margarine, and being margarine could not be sold at all if it contained more than 10 per cent. of butter fat. The answer was that, looking at the Act of 1899, it was quite plain that the Act did contemplate certain things which, according to this argument, ought to be called margarine, and yet which were called by a different name. In Section 1 of the Act, "adulterated or impoverished butter (other than margarine)" was referred to as one of the articles which was not to be imported except upon certain conditions. It would follow, if Mr. Danckwerts' argument was right, that if impoverished butter was imported it could only be sold as margarine, and that it could not be sold at all if it contained more than 10 per cent. of butter fat. That was the logical result of the argument. The facts as to the substance in question in the case were perfectly well known. It was butter mixed with milk, and it could not be sold as butter because of Section 6 of the Sale of Food and Drugs Act, 1875. But it might be sold under that Act with a proper protective label. Now, it was contended that it could not be sold at all because it contained more than 10 per cent. of butter fat, and had been made "margarine" by Act of Parliament. He thought it was butter and milk mixed together, and he found nothing to compel him to say that butter and milk mixed was not to be sold except as margarine, or, what was more important, was not to be sold at all. He thought the magistrates were perfectly right; the substance was not margarine, but butter and milk.

Mr. JUSTICE DARLING and Mr. JUSTICE CHANNELL delivered judgment to the same effect.

AMOUNT OF WATER IN MARGARINE.

JOSEPH BURTON AND SONS (LIMITED) *v.* MATTINSON.

This was an appeal by way of case stated from a decision of justices convicting the appellant company, under Section 6 of the Sale of Food and Drugs Act, 1875, of selling margarine not of the nature, substance, and quality demanded by the purchaser.

The facts were as follows: On December 9, 1901, the appellants exposed for sale at their shop at Rushden, Northamptonshire, a substance labelled margarine and ticketed for sale at 6d. per pound. The respondent entered the shop and called for 1 pound of margarine, to be supplied out of the margarine so exposed for sale, and he was served with 1 pound of the substance, for which he paid 6d. The formalities required by the Act were carried out, and the substance was duly analysed. It was admitted that the appellants had complied with all the requirements of the law as to labelling and selling the substance sold as margarine. The analyst's certificate stated that the sample contained the percentage of foreign ingredients as follows: "Water, 21 per cent. (and this was at least 5 per cent. in excess of the amount of water which margarine should contain.)" It was objected on behalf of the appellants that the words in brackets were no proper part of the analyst's certificate, but were a mere expression of opinion, which was in no way receivable in evidence. It was stated by the analyst, who was called as a witness for the respondent on the requisition of the appellant, that the average percentage of water contained in margarine was from 8 to 10 per cent., so that, in stating in his certificate that this sample contained at least 5 per cent. in excess of the maximum amount which should be present in margarine, he was dealing with the margarine leniently and allowing the same maximum as is allowed in butter—namely, 16 per cent.; that margarine, in his experience, should contain rather less moisture than butter; that well-made butter contains 10 to 12 per cent. of water on an average; that the principle or valuable constituent of butter was fat, of which the percentage should be from 80 to 85; that the only valuable constituent of margarine was also fat, of which it should contain at least 85 per cent.; that this particular sample contained only 70 per cent. of fat, so that what was lacking in fat was made up in water and salts; that margarine was made from various fats, either animal or vegetable—it was usually made from the more liquid portions of animal fat mixed with various vegetable fats; that margarine should, in his opinion, imitate butter, not only in appearance, but also in its constituent elements. It was admitted that there was nothing in the substance sold injurious to health, and that margarine was sold at times in the district for as much as 8d. to 10d. per pound, and for as little as 4d. per pound, so that 6d. per pound was the price of a comparatively cheap quality of margarine. It was admitted by the two witnesses of the respondent that the sample sold was in outward appearance an imitation of butter. No evidence was given on the part of the appellants, but it was contended by counsel for the appellants on the above facts that the appellants had committed no offence in point of law, because (1) the term "margarine" was not a conventional term or one affixed by usage to any substance, but was a statutory term affixed by the Margarine Act, 1887, to all substances, whether compounds or otherwise, prepared in imitation of butter, and that the substance then in question being prepared in imitation of butter was rightly sold as margarine, and was, therefore, of the nature and substance demanded by the purchaser; (2) the quality of the substance supplied was that demanded by the purchaser, being the margarine at 6d. per pound then exhibited for sale in the appellants' shop at Rushden; (3) the Legislature had not fixed any standard for margarine nor enacted of what ingredients it should be composed nor the proportions in which they should be combined; (4) if it be necessary that a substance sold as margarine should imitate butter, not only in outward appearance and nature, but in the ingredients used in its composition, the substance then in question, being not only similar to butter in such outward appearance, but also being composed of the same ingredients as butter—namely, fat, water, and salt—was margarine, and an article of the nature, substance, and quality demanded. It was contended for the respondent that, although the Legislature had not fixed any standard for margarine, neither had it fixed any standard for butter, and yet there had been several convictions for selling butter which contained more than 16 per cent. of water, and such convictions had been upheld on appeal, and that if it was illegal to sell butter with more than 16 per cent. of water, it was also illegal to sell margarine with more than the same percentage. That if it was lawful to sell as margarine a substance containing 21 per cent. of water, there was no reason why such substance should not be sold containing 40 or 50 per cent. of water. That water was not a substance prepared in imitation of butter within the definition of margarine contained in the Margarine Act, 1887. The justices overruled the appellants' objections, being of opinion that the water found in the margarine was excessive, and that, therefore, the article sold was not of the nature, substance, and quality demanded, the same being adulterated with water, and they convicted the appellants subject to the present case, imposing a penalty of £1 with costs.

Mr. Avory, K.C., and Mr. W. H. Stevenson appeared for the appellants; and Mr. Swinburne-Hanham for the respondent.

The court dismissed the appeal yesterday.

The Lord Chief Justice said that in this case the magistrates, the sale being a sale of margarine, and the provisions of the Margarine Act, 1887, having been complied with, had found that the water found in the margarine was excessive, and that, therefore, the article sold was not of the nature, substance, and quality demanded, the same being adulterated with water. That meant that the substance was margarine and water, and not margarine. The Court had been pressed to say that this finding could not stand, because there was no evidence on which the magistrates could come to the conclusion that the substance was margarine and water. It was contended that the whole matter was governed by Section 3 of the Margarine Act, 1887, which enacted that "the word 'margarine' shall mean all substances, whether compounds or otherwise, prepared in imitation of butter, and whether mixed with butter or not, and no such substance shall be lawfully sold except under the name of margarine and under the conditions set forth in this Act." It was contended that, if a person sold any imitation of butter and called it margarine, there would be practically no inquiry into the nature of the substance. The objection to this contention was that it overlooked the provisions of the legislation under which the purchaser is entitled to get what he asks for. He did not say that the presence of any particular percentage of water prevented an article from being margarine. But the court had merely to see whether there was any evidence that the article in question was not margarine but margarine and water. The analyst's certificate said that the substance contained 21 per cent. of water, "and this was at least 5 per cent. in excess of the amount of water which margarine should contain." Unless the analyst was called the certificate was, under the Act, sufficient evidence of the facts therein stated. The defendant could, however, require the analyst to be called. In this case this was done, and the analyst in his evidence stated the line of reasoning which led him to the conclusion that he was right in stating that margarine should not contain so large a percentage of water as was present in this case. The analyst's reasoning had been criticised. It was said he ought not to have compared margarine with butter. But no evidence had been called on the point; and it was impossible to say that the magistrates were not justified in relying on the certificate supplemented by the analyst's evidence, and in holding that the substance was margarine and water and not margarine.

Mr. Justice Darling and Mr. Justice Channell delivered judgments to the same effect.

MILK OF INFERIOR QUALITY FROM A SINGLE COW.

KING'S BENCH DIVISION.

(Before the LORD CHIEF JUSTICE OF ENGLAND, MR. JUSTICE DARLING, and MR. JUSTICE CHANNELL.)

SMITHIES *v.* BRIDGE.

From the "Times" of May 3, 1902.

THIS was an appeal from the Chelmsford Quarter Sessions. The case raised an important question as to whether a person supplying milk exactly as it came from a cow could be convicted of an offence against Section 6 of the Sale of Food and Drugs Act, 1875, if the milk was of inferior quality. The following facts appeared from the case stated.

The appellant, William Smithies, of Clacton-on-Sea, was convicted at Thorpe le Soken Petty Sessions, under Section 6 of the Sale of Food and Drugs Act, 1875, for selling to the respondent on August 15, 1901, to the prejudice of the purchaser, milk not of the nature, substance, and quality demanded, and he was fined £20. Smithies appealed to Quarter Sessions, when the following facts were proved: The respondent asked to be supplied with a pint of new milk, and on analysis it was found to contain only 2·09 per cent. of fat. The milk had not been tampered with or adulterated in any way, but was in the same condition as when it came from the cow. The small percentage of fat was caused by there being a long interval of sixteen hours between the morning milking, at which the milk in question was drawn from the cow, and the previous milking. The owner of the cows had been told by veterinary surgeons that this system of milking, while it increased the quantity of milk at the morning milking, caused it to be deficient in fat, as the latter became absorbed. The cows were not the property of the appellant, but of Mr. Thomas Lilly, from whom the appellant purchased the milk under a guarantee, but the appellant did not rely upon this guarantee as a defence. The justices at the Quarter Sessions were of opinion that there had been no adulteration of or abstraction from the milk, but that, nevertheless, the offence charged had been proved. They confirmed the conviction, and reduced the fine to £1.

MR. WARBURTON, for the appellant, said that as this was milk exactly as it came from the cow, it was pure milk, although of inferior quality, and therefore the appellant could not be convicted—"Hoyle *v.* Hitchman" (4 Q.B.D., 233) and "Morgan *v.* Auger" (L.J. Newsp. for April 26, 1902, p. 228).

MR. C. E. JONES, for the respondent, submitted that it was a question of fact for the justices—"Hewitt *v.* Taylor" (1896, 1 Q.B., 287), "Dyke *v.* Gower" (1892, 1 Q.B., 220). He referred to the Sale of Milk Regulations, 1901, made by the Board of Agriculture, after the date of the alleged offence, prescribing that where milk contained less than 3 per cent. of fat it should be presumed till the contrary was proved that it was not genuine.

MR. JUSTICE CHANNELL, in giving judgment, said that the court were not agreed as to the result, but he was not sure that there was any large difference between them as to the principles in question. It was material to recollect that the prosecution was under Section 6, under which it had been clearly decided that there was no question of guilty knowledge or fraudulent intent. The question merely was whether the person charged had sold something different from, and inferior to, the thing demanded by the purchaser. The case of "Goulder *v.* Rook" (1901, 2 K.B., 290) brought out these propositions. That was a case of beer, which was a manufactured article, but in the case of a natural product, such as milk, the same principles were applicable. If a person asked for new milk and got something which was not that which he would expect to get as new milk, the offence had been committed. If in ordinary cases the person charged was able to prove that what he had sold as milk was the identical thing, without adulteration and without manipulation, that came from the cow, then, unless there was something further, he clearly proved that the substance sold was milk. If the man did not know, and made inquiries and discovered that the cow had some illness, that would confirm the analysis, and go to show that the thing sold was not of the nature, substance, and quality of the article demanded. He had sold something which was the direct product of the cow, but in the case his Lordship was putting it was a cow which was not producing milk, but another liquid, and on those facts the seller, though perfectly innocent, ought to be convicted under this section. One had to apply those principles to the present case. The view of the magistrates was that there was no reason for suspecting any mistake in the analysis, and that it showed that there was a smaller quantity of fat than even the poorest kind of milk contained, and that, by reason of the cow being milked at unusual times, the substance that came from the cow was not a substance that could properly be called milk. That seemed to him to be a question of fact, and if it was not milk, it was not of the nature, substance, and quality demanded. It was not for the court to interfere with a conclusion of fact. This might be a hard case, but, if they did not lay down a rule such as he had done, it might do mischief in other cases.

Mr. JUSTICE DARLING said that he was very sorry that he did not agree. The purchaser asked for new milk, and he got milk exactly as it came from the cow. What he got was undoubtedly new, and the question was whether it was milk. In ordinary language it was milk, and it was exactly in the condition in which the cow gave it. If a man asked for milk, he meant cow's milk, but not the milk of a diseased cow. Seeing that it was food, he must be understood to demand milk fit for human consumption. If it was not milk, what was it? Some cows gave richer milk than others; and, there being no standard, the appellant supplied what he was perfectly entitled to describe as milk. If this was only a case of milk, he should not trouble to differ from the other judges, but he feared that the decision might be applied to some other natural product as to which there was no standard—*e.g.*, apples from an apple-tree which was sending the malic acid into its roots, although in so preposterous a case his brother might be able to distinguish it. It was a principle capable of dangerous application to say that a man could be convicted where, without any element of fraud, he sold a natural product in the state in which it was produced.

The LORD CHIEF JUSTICE said that he could not distinguish in this case between Lilly and the appellant. The magistrates had dealt with it as a question of fact, and, if there was evidence on which they could come to the decision they did, the court ought not to overrule it. For the reasons given by his brother Darling, he came to the conclusion that it was not possible to distinguish between the appellant and Lilly; and the only question was whether there was evidence that the article sold was not of the nature, substance, and quality demanded. The milk was deficient in 30 per cent. of the fat proper to genuine milk. If there had been evidence that the milk came direct from the cow, and no evidence of an abnormal condition of things, it would have been wrong to convict. But in this case it was found that the condition of the milk was due to the way in which the cows had been milked—*i.e.*, there had been an abnormal interval of sixteen hours. The magistrates had found that it was so deficient in fat that it could not fairly be described as milk. He did not say that he would have come to the same conclusion, but it was impossible to say that there was no evidence that it was not ordinary new milk. He did not wish to say that there was any arbitrary standard. The order of the Board of Agriculture did not establish a standard for what was or was not genuine, but only *primâ facie* evidence. It could still be proved by the defendant that the milk was genuine. If it turned out that the article produced was the result of an abnormal condition of things, whether from disease or from abnormal treatment, there was evidence on which the justices might find that the article was not of the nature, substance, and quality demanded. The purchaser, when he asked for new milk, was entitled to have an article which was not other than ordinary new milk by reason of any special way in which the cows had been treated.

The appeal was accordingly dismissed, but without costs.

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VOLUME XXVII.—No. 315.
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THE ANALYST.

JUNE, 1902.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE monthly meeting of the Society was held on Wednesday evening, May 7, in the Chemical Society's Rooms, Burlington House. Mr. W. W. Fisher, M.A., in the absence of the President, occupied the chair.

The minutes of the previous meeting were read and confirmed.

Certificates of proposal for election to membership in favour of Messrs. M. Wynter Blyth, B.A. (Cantab.), B.Sc. (London), F.I.C., and W. F. Sutherst, Ph.D. (Geneva), A.I.C., were read for the second time; and a certificate in favour of Mr. Alfred Howard, A.R.C.S., A.R.S.M., analytical chemist, 186, Stanstead Road, Forest Hill, S.E., Gas Examiner to the London County Council, and to the Lewisham Borough Council, was read for the first time.

Messrs. W. H. Jollyman, J. T. Norman, J. Golding, and W. Bowen were elected members of the Society.

The following papers were read: "The Composition of Milk," by H. Droop Richmond; "Fluorides as Butter Preservatives, with Observations on their Influence on Artificial Digestion," by Otto Hehner and C. W. Hehner; "Note on a convenient Apparatus for the Chemical and Bacteriological Examination of the Atmosphere," by Wm. Chattaway and F. M. Wharton; and a "Note on some Drug Standards," by H. Wippell Gadd.

FLUORIDES AS BUTTER PRESERVATIVES, WITH OBSERVATIONS ON THEIR INFLUENCE ON ARTIFICIAL DIGESTION.

BY OTTO AND CHARLES W. HEHNER.

(*Read at the Meeting, May* 7, 1902.)

THE evidence taken by the Departmental Committee on the use of preservatives and colouring matters in food contains only one very brief reference to fluorides, and that of a negative character, one witness being asked whether he had met with any article of food containing fluorides, the reply being that he had not met with them, but had heard of their use.

During the last few months there appeared on the London markets a good deal of Brittany butter which either did not contain any boric preservative at all (while the Brittany practice had hitherto been to add from 0·4 to 0·5 per cent. of boric acid), or only unusually small quantities ; yet these butters possessed the keeping qualities of butter containing preservatives. Our attention was directed to these butters by a firm of the highest standing in the butter trade. After searching for salicylic, benzoic, and sulphurous acids, with negative results, we succeeded in obtaining in a few, but by no means all such instances, distinct evidence of the presence of a soluble fluoride. Fifty grammes of the butter were melted in a platinum basin, the aqueous layer separated from the fat, rendered slightly alkaline with sodium hydroxide, evaporated, the residue ignited, transferred to a platinum crucible, strong sulphuric acid added, and the crucible covered with a waxed slip of glass upon which a distinguishing mark had been scratched with a pin. The crucible and contents were gently heated upon a sand-bath for two hours, and the glass slip examined after the removal of the wax. We found that 1 milligramme of calcium or sodium fluoride showed distinct etching of the glass.

Instead of melting the butter in a platinum basin, the melting out can be done equally well in glass, but a little alkali should be added.

It soon became apparent that in the presence of boric preservative fluoride could not thus be discovered, for in presence of both strong sulphuric acid evolves boron fluoride. By the use of dilute sulphuric acid the formation of boron fluoride is avoided, and as long as the amount of boric acid is not greater than five times that of the fluoride, a reaction (etching of the glass) can be obtained. But we found it far preferable to get rid of the boron compounds before testing for fluorides. The aqueous liquor is separated from 50 grammes of the butter, and without clarifying it calcium chloride is added, the liquid heated to boiling, and a small excess of sodium carbonate used to precipitate the calcium compounds. The precipitate consists of calcium borate, fluoride, carbonate, with some phosphate and perhaps sulphate. It is filtered off, burnt and treated with hot dilute acetic acid ; this dissolves out the carbonate, borate and phosphate. The residue is again collected on a filter, ignited, and treated with strong sulphuric acid. Distinct etching of the glass results in presence of fluoride. We have thus often been able to get fluorine reactions when, without separation of borate, the tests were quite negative. We also endeavoured to make this test a quantitative one, though with no conspicuous success. The final precipitate containing the calcium fluoride was weighed after the etching process, the sulphuric acid was driven off, the residue strongly ignited, and the weight of the resulting calcium sulphate again ascertained. Since calcium fluoride, with its molecular weight of 78 yields 136 parts of calcium sulphate, the increase of weight should furnish the measure of the amount of fluorine. But even in cases where no etching could be observed, and where we must presume fluorides to have been absent, there was a distinct residue left after the acetic acid treatment, and this, in most cases, slightly increased in weight after being converted into sulphate. It is probable that the ignited calcium phosphate was rather more insoluble in the dilute acetic acid than was expected, and tricalcium phosphate, on ignition with strong sulphuric acid, we have satisfied ourselves, gains notably in weight, the ignited residue corresponding in composition with a mixture of (calcium sulphate) and calcium pyrophosphate with a little metaphosphate. But when a fluorine reaction was obtained, the increase in weight was nearly always materially higher than in the negative cases. The increase in weight corresponded from about 0·04 to 0·08 per cent. of sodium fluoride. This amount is probably overestimated by about 0·02 per cent., so that the actual quantity of sodium fluoride present is from 1 to 4 grains per pound. These figures make no pretence to accuracy, but cannot be far off the truth.

We obtained some of the fluoric butter preservative from Brittany. This, on analysis, was found to contain about 98 per cent. of sodium fluoride, the remainder being moisture. A strong trace of lead, doubtless due to the use of lead vessels in the preparation of the hydrofluoric acid, was also present.

With the precipitate obtained from some of the samples of butter several glass plates in succession could be strongly etched.

We have so far found fluoride in twenty samples of butter; in ten samples there was no boric preservative; while in ten others this was also present in amounts varying from 0·18 per cent. to 0·36, or from 12·6 to 25·2 grains per pound. We have also searched for fluoride in a few samples from other countries in which circumstances appeared to render the presence of some preservative other than boric likely, but with negative results. So far the use of fluoride seems to be confined to certain French butters.

As traces of fluorine must be present, and are stated to have been found, in milk, we tested the ash of 50 grammes of milk, but with quite negative results. The reactions obtained with 50 grammes of butter must therefore be due to the added material.

The Preservatives Committee of the Local Government Board recommended that borate be the only permitted preservative in butter and margarine. As, however, it might be argued that no evidence as to the use of fluorides in butter was before the Committee, the recommendation in question might not apply to that preservative, it becomes important to inquire into the physiological action of fluorides when taken in the quantities that might be consumed in butter. Taking the maximum amount we have met with—namely, about 4 grains to the pound—it would follow that in 2 ounces of butter, which a person might very easily consume, ½ grain of sodium fluoride would be contained. We take from the American Dispensatory (17th edition, p. 1634) the following statements: "Waddell states that the alkaline fluorides are not pronounced irritants, and when taken internally in doses of a grain to a grain and a half continuously they reduce the force and the frequency of the pulse, at the same time depressing the temperature and increasing somewhat the flow of urine, but not distinctly affecting either the respiratory or the cutaneous functions. This accords with the physiological studies of Tappeiner, who found in animals the soda-salt to powerfully depress blood-pressure by acting on the vaso-motor centres. Death, after profound collapse, was produced by centric failure of respiration. Dr. Waddell also [illegible] that there is an enormous decrease in the number of red blood corpuscles,
he was afra[illegible] s, but does not prove, to be the result of a direct action upon the
mittee had reco

permitted in the ca raise, to say the least, a presumption that the quantities of
constituents causing butter preservatives are not without injurious action upon
case of cream, which ber of experiments we have made with digestive ferments

show clearly that fluorides have, in some cases at least, a powerful inhibitory action. As regards some fermentative actions this is already well known. Thus, in Oppenheimer's book on "Ferments," translated by Mr. C. A. Mitchell, we find the statement that sodium fluoride prevents the action of rennet (p. 144), and that fluorides have a marked injurious effect upon the zymase obtained by Buchner from yeast (p. 242). In Effront's method for the suppression of bacterial organisms in the manufacture of alcohol, it has been found (Maerker, "Das Fluss-saeureverfahren in der Spiritusfabrikation," Berlin, 1891) that as little as 10 grammes of ammonium fluoride in 100 litres of water may injure the germinating power of barley, and that more than 3 grammes in 100 litres may interfere with the diastatic power of malt.

Now, since butter with about 13 per cent. of water was found to contain from 0·02 to 0·06 per cent. of fluoride, calculated as the sodium salt, the samples contained an aqueous solution of from 0·16 to 0·46 per cent. strength, which solution inhibits the growth of micro-organisms in the butter. In the experiments which we are about to give a more dilute solution was employed in every case.

Ptyalin Experiments.—Ten grammes of air-dry arrowroot were gelatinized with hot water and the bulk made up to 250 c.c.; 5 c.c. of sublingual saliva were added when the solution had a temperature of 38° C. After one hour at that temperature iodine gave only a brown coloration. The cupric reducing power corresponded to 42·63 per cent. of maltose calculated on the original starch taken.

A precisely similar experiment was made at the same time, 0·1 gramme of ammonium fluoride (containing 55·8 per cent. of fluorine) being added after the arrowroot had been gelatinized. After an hour's digestion iodine gave a strong blue colour; the solution had no cupric reducing power.

A similar experiment was made with an equivalent quantity of sodium fluoride. After one hour's digestion the solution gave a blue colour with iodine and did not reduce Fehling solution.

It follows that salivary action is prevented by a solution containing 0·04 per cent. of sodium fluoride, or its equivalent in ammonium fluoride.

Peptic Digestion.—Three lots of 50 grammes each of hard-boiled white of egg, which had been rubbed through a fine sieve, were heated to 40° C. with 250 c.c. of a 0·2 per cent. HCl solution containing enough pepsin to dissolve the whole of the white of egg. One lot did not receive any fluoride, the second 0·1 gramme, and the third 0·05 of ammonium fluoride. After five hours the whole of the white of egg in solution 1 had dissolved, the amount of egg had not visibly diminished in solution 2, while a very large proportion was left undissolved in sample 3.

It is clear that as little as 0·02 per cent. solutions of fluoride greatly interfere with peptic digestion. The difference in the appearance of the contents of the test-bottles was so striking that quantitative experiments seemed unnecessary.

Rennet Experiments.—Contrary to the statement in Oppenheimer's work, as [illegible] above, we could not perceive any interfering action of a small amount of [illegible] the milk-curdling action of a solution of rennet. Nor was such in[illegible] be expected with quantities of fluoride smaller than amounts en[illegible] the calcium salts contained in the milk. As soon, however, as [illegible] was overstepped, the fluoride stopped the rennet action.

Diastatic Action.—The proportions of starch (arrowroot) and water were the same as in the saliva experiments, but instead of the saliva 25 c.c. of a cold water infusion of malt were used, the temperature being maintained at 65° C. for one hour. The iodine reaction after that time was markedly different in the fluids with and without fluoride; in the former case only a darkening in the colour was the result, while in the latter a strong erythrodextrine reaction was always observed. The reducing power, calculated in percentages of maltose upon the weight of the arrowroot taken, was found to be as follows:

Without ammonium fluoride.		With ammonium fluoride.
67·4	... ...	57·4
62·1	... ...	45·0

These figures bear out Maerker's warning previously referred to. At the same time, it appears that the injurious action of fluorides upon the salivary starch-solvent action is far more pronounced than in the case of the corresponding malt diastatic effect. From some qualitative tests made by us it also appears that the inhibitive influence of fluorides upon the ferment, which forms mustard oil from myrosin, is but slight.

Against the use of fluorides in the manufacture of alcohol, as in Effront's process, no objection can be raised from a sanitary point of view, because the chemical does not enter into the final product. The case is far otherwise when the fluoride is directly admixed with food materials and is consumed with them. At present the use of fluoride, as far as our experience goes, appears to be confined to a single country—France. We hope that our remarks may be the means to arrest the further spread of its employment.

We may further take this opportunity to express the hope that the President of the Local Government Board will no longer delay the issue of regulations concerning the use of preservatives, the Committee's report having been presented nearly six months ago.

DISCUSSION.

Mr. LLOYD said that about fifteen years ago he [illegible] one of the colonies that a means of preserving butter [illegible] could not be detected by analysis, and, after [illegible] fluorides were being used. He had reported that fluorides [illegible] than some other preservatives, and, as far as he knew, [illegible], of boracic acid. Last year, however, he had been [illegible] in the form of sodium fluoride, were again being [illegible] up as soon as the possibility of the prohibition [illegible] his. became known, the material was introduced [illegible] the he was afraid that its use might not be [illegible] obtain mittee had recommended that the use of [illegible] d-point, permitted in the case of butter—a substance [illegible] d greatl constituents causing decompos [illegible] ng the case of cream, which was [illegible] arge substances liable to decomposition, instead of being washed out, were all contained in it. So far as he knew, sodium fluoride was being used—or, at any rate, was ready to be used—not merely for butter, but for milk and cream. He thought that a debt of gratitude was due to the authors of this paper for having described so clearly a method by which it could be detected. It had taken him a very long time, in years gone by, to detect the preservative in the colonial butter he had referred to, and if he had not been perfectly certain that some preservative was present, he might have considered the search hopeless, and have said that no preservative was present. In regard to the statement that the action of fluorides upon rennet was only marked when sufficient was present to neutralize or combine with the lime present in the sample of milk experimented with, he would point out that the action of rennet was a time action, and the question was not whether the action might be actually stopped, but whether it might not be delayed; and he would like to hear whether the authors had made any experiments which would show whether the action of rennet was delayed to any extent by quantities of fluorides too small to stop it altogether, for he fancied that the digestion of milk in the infant must be to some extent similar to the action of rennet, and therefore that a substance added in small quantities, if it delayed the action of rennet, would probably also delay digestion in the infant, and would, on that account, be injurious, although it might not actually prohibit entirely the action of rennet.

Mr. Allen said that probably it was within the knowledge of the authors and of others present that the use of silico-fluoride of sodium as a preservative had been patented, and that the compound was manufactured to a considerable extent at Warrington. The use of sodium fluoride had probably grown out of it. It was interesting, seeing that the association of boric acid with fluorides had been mentioned, to note that boron compounds also were manufactured largely at Warrington. He would like to hear if the authors had any suspicion that the two were used in conjunction. Possibly the boron was in the form of boro-fluoride of sodium ($NaBFl_4$); or borax and hydrofluoric acid might be used, and would probably form a compound ($HBFl_4$) eminently suitable for the purpose in question.

Mr. Richmond mentioned that it had been proposed to use fluo-boracic acid as a preservative, and some years ago he had examined a sample which contained it. He did not think, however, that it had ever been extensively used.

Mr. Briant said that sodium fluoride had been patented some five or six years ago as a preservative for beer, but in that case, again, no practical application had followed.

Mr. Fisher said that he remembered that some years ago potassium boro-fluoride had been recommended as a strong and powerful antiseptic and an excellent preservative, and he had made a few experiments with it at the time, but he had never met with it professionally, and did not know that it had come into use.

Mr. O. Hehner, in reply, said that their experiments had been confined to ascertaining the presence of fluorides, and he could not say whether boro-fluoride had been also used or not. In many cases boro-fluoride was certainly absent, for they had found fluoride in the absence of boron. He doubted whether at the present time any Australian butter contained fluorides. Their experiments in regard to the action of rennet were quite qualitative. The results obtained had been that without fluorides coagulation took place in a very short time at blood heat, while after the addition of fluorides the milk remained fluid for a long time. The inhibitive action of the fluoride was only apparent after a certain quantity had been added. The first few drops of the solution, which made no difference, were, he surmised—though he had not actually determined whether such was the case—necessary for the precipitation of the calcium. Their remarks had been intended mainly for the information of analysts, but partly also for the information of the Board of Agriculture. He presumed that the limitations recommended by the Departmental Committee on Food Preservatives could only be regarded as extending to those preservatives which had been referred to in the course of the Committee's inquiry. Only such substances could be prohibited or permitted as were known to be used at the time of the inquiry. As, however, formal regulations had not yet been issued, there might be still time to extend the prohibition to fluorides, which, in his opinion, were injurious.

THE RAPID ESTIMATION OF BORIC ACID IN BUTTER.

By H. Droop Richmond, F.I.C., and J. B. P. Harrison, F.I.C.

(*Read at the Meeting, April* 9, 1902.)

The report of the Preservatives Committee recommending a limit of 0·5 per cent. of boric acid in butter has rendered it desirable to make the estimation of this substance as simple and rapid as possible. We have been in the habit of using a slight modification of Thomson's method: About 25 grammes of butter is weighed into a stoppered cylinder, enough water to make with the water already present a number of c.c. equal to the weight of butter in grammes, and 10 to 15 c.c. of chloroform are added; the contents of the cylinder are warmed, mixed, and allowed to separate; an aliquot portion of the aqueous solution is drawn off (each c.c. containing the boric acid of 1 gramme of butter), made alkaline, evaporated, ignited, and the soluble ash dissolved in hot water. This solution is made neutral to methyl orange, boiled to expel CO_2, and, after the addition of glycerol, titrated with standard alkali. We have proved the accuracy of this method, have found that the insoluble ash is free from boric acid, and have also ascertained that the ignition cannot be dispensed with, the results without it being from 0·05 to 0·10 per cent. too high.

The evaporation, ignition, and extraction of the ash with hot water take up some little time, and we experimented to find a method which would avoid this. Our first experiments were with a slight modification of Deniges' method for the estimation of boric acid in milk (*J. Pharm. Chim.*, vi. (2), 49), but we failed to obtain sufficiently concordant results, due, as we soon found, to the fact that the end-point, when titrating boric acid with phenolphthalein in milk-sugar solution, varied greatly with the temperature. We found, however, that when the solution was boiling the end-point was constant and sufficiently sharp. The end reaction is distinctly sharper in milk-sugar solution than in water; a 5 per cent. solution of milk-sugar was somewhat arbitrarily adopted.

Our preliminary experiments were made with a weighed quantity of boric acid dissolved in 10 c.c. of water, to which was added 15 c.c. of a 10 per cent. milk-sugar solution and 3·25 c.c. of water (the average amount contained in 25 grammes of butter). A few drops of phenolphthalein were added, and the solution raised to the boiling-point; $\frac{N}{2}$ caustic soda solution was added till a faint but distinct pink colour was observed; about 15 c.c. of glycerol was then added, and the solution titrated until a pink colour was produced.

The following results were obtained:

Boric Acid taken.	C.c. of $\frac{N}{2}$ Soda in Milk-sugar Solution.	C.c. $\frac{N}{2}$ Soda in Glycerol Solution.	Difference.	Boric Acid found.
0·100	0·53	3·23	2·70	0·099
0·070	0·33	2·24	1·91	0·070
0·050	0·26	1·62	1·36	0·050
0·020	0·10	0·65	0·55	0·020

It is seen that the difference between the two titrations indicates 84·4 per cent. of the total boric acid, and 1 c.c. of $\frac{N}{2}$ soda is equal to 0·0368 grammes of boric acid.

The influence of slight dilution was studied; a 1 per cent. boric acid solution was used.

Ten c.c. boric acid solution + 15 c.c. 10 per cent. milk-sugar solution + 0 c.c. water, indicated 0·100 gramme boric acid.

Ten c.c. boric acid solution + 15 c.c. 10 per cent. milk-sugar solution + 3·25 c.c. water indicated 0·100 gramme boric acid.

Ten c.c. boric acid solution + 15 c.c. 10 per cent. milk-sugar solution + 7·5 c.c. water indicated 0·098 gramme boric acid.

The dilution due to the extreme percentages of water likely to be met with in butter will not affect the determination.

Various substances which may occur in butter were added to the boric acid solution.

Ten c.c. boric acid solution + 15 c.c. 10 per cent. milk-sugar solution + 4 c.c. milk indicated 0·100 gramme boric acid.

Ten c.c. boric acid solution + 15 c.c. 10 per cent. milk-sugar solution + 3·25 c.c. of a solution containing butyric and caproic acids indicated 0·100 gramme boric acid.

Ten c.c. boric acid solution + 15 c.c. 10 per cent. milk-sugar solution + 3·25 c.c. of a solution containing 0·030 gramme P_2O_5 indicated 0·102 gramme boric acid.

Ten c.c. boric acid solution + 15 c.c. 10 per cent. milk-sugar solution + 3·25 c.c. of a solution containing 0·059 gramme P_2O_5 indicated 0·102 gramme boric acid.

It is thus seen that none of the substances likely to be present in butter will affect the determination. Considerable quantities of phosphoric acid have, however, a prejudicial effect; a solution free from boric acid, and containing 0·297 gramme P_2O_5, indicated 0·019 gramme of boric acid.

Mixtures of boric acid and borax may be titrated by adding a little mineral acid to liberate boric acid from the borax.

An amount of commercial preservative equal to 0·100 gramme of boric acid dicated 0·103 gramme boric acid.

As the result of our experiments, we have adopted the following method: eigh out 25 grammes of butter in a beaker, add 25 c.c. of a solution containing grammes of milk-sugar and 4 c.c. N sulphuric acid to 100 c.c. Place in the water-en till the fat is just melted and stir well; allow the aqueous portion to settle for a w minutes and draw off 20 c.c.; add a few drops of phenolphthalein, bring to the il, and titrate with $\frac{N}{2}$ soda till a faint pink colour just appears; add 12 c.c. glycerol, d titrate till a pink colour appears. The difference between the two titrations, less e amount of alkali required by 12 c.c. of glycerol, multiplied by 0·0368, will give e amount of boric acid in 20 c.c., and this, multiplied by

$$\frac{100 + \text{percentage of water}}{20},$$

ill give the percentage. If the percentage of water is about the average, it may be ken as 13 without appreciable error.

Generally, the number of c.c. of $\frac{N}{2}$ soda used, multiplied by 0·2, will very closely proximate to the percentage of boric acid.

The following test experiments will show that the method is accurate enough r all purposes. They were made by weighing out 25 grammes of butter free from ric acid, adding a known weight of boric acid or a commercial preservative of own boric acid value, and estimating as already described:

Boric Acid added. Per Cent.	Boric Acid found. Per Cent.	C.c. $\frac{N}{2}$ Soda $\times 0·2$
0·10	0·11	0·11
0·25	0·26	0·25
0·35	0·36	0·35
0·45	0·46	0·44
0·56	0·55	0·52
0·62	0·60	0·58
0·88	0·86	0·84

Butters free from boric acid have shown a difference between the two titrations ual to the amount of alkali required by the glycerol. We communicated the ethod to Mr. Hehner, and at his request Mr. B. W. J. Warren has sent the llowing results to us:

Boric Acid added. Per Cent.	Boric Acid found. Per Cent.
0·48	0·50
0·60	0·59
0·67	0·70
0·47	0·46
0·11	0·13

Mr. Warren also found in another butter 0·45 per cent. in daylight, and 0·44 and 42 per cent. by gaslight.

DISCUSSION.

Mr. B. W. J. **WARREN** said that one difficulty he had met with in his experiments referred to in the paper was the production of a pink colour, which rendered one very liable to overshoot the mark in titrating. He had found, however, from some investigations of Mr. R. T. Thomson, that lacmoid would answer a similar purpose to methyl orange, and he had accordingly experimented with lacmoid, and had obtained very good results. The lacmoid could be easily seen in the turbid solution obtained from the butter.

Mr. F. J. **LLOYD** inquired why a solution of milk-sugar was used. He had made estimations simply by extracting with boiling water and filtering, and had found no difficulty in obtaining accurate results without using a solution of milk-sugar—simply adding the sulphuric acid and boiling.

Mr. **RICHMOND** hoped that the very interesting information which Mr. Warren had given with regard to the use of lacmoid, which was certainly more convenient to use than phenolphthalein, would be embodied not merely in a short note in this discussion, but in a paper before the Society, going more fully into the details of the method, and giving the results obtained. Mr. Warren's method appeared, as far as he could see, to be better than their own, and he should try it at once. Before answering Mr. Lloyd's question, he would like to hear exactly how Mr. Lloyd obtained the result he mentioned with water—whether he titrated, first of all, with methyl orange and then with phenolphthalein and glycerol, or simply, as they had, first titrating, till the pink colour appeared, with water, then adding glycerol, and going on with the titration and taking the difference.

Mr. **LLOYD** said that the titration was made first with methyl orange, and then with phenolphthalein and glycerol, methyl orange being used before the addition of the glycerol.

Mr. **RICHMOND** said that in that case he could hardly understand Mr. Lloyd getting accurate results, because there must be a certain amount of casein and other proteids in the aqueous portion, as well as a certain amount of phosphoric acid in the form of phosphates. These two both interfered when methyl orange and phenolphthalein were both used, because the end-points for methyl orange and for phenolphthalein were very different. That was the reason for stating in the paper that evaporation and ignition could not be dispensed with, the results otherwise being from 0·05 to 0·1 per cent. too high. The reason for using milk-sugar was that they had found that with milk-sugar, in boiling solution, the end-point was sharper than was the case in an aqueous solution simply. The true reason was that the amount of dissociation was more sharply determined in the milk-sugar solution than in the aqueous solution, the practical result being that the end-point was easier to see.

A CONTRIBUTION TO A KNOWLEDGE OF THE CHEMISTRY OF CIDER.

BY ALFRED H. ALLEN.

(Read at the Meeting, December 11, 1901.)

CIDER is well known to be prepared by the alcoholic fermentation of apple-juice. Skilful manufacturers maintain that the juice itself is all that is needed for the production of a perfect article, the processes being of the simplest kind:—namely, the preparation of the juice by pressing the sliced or grated apples; exposure of the juice at a temperature of or near 60° F., when it undergoes spontaneous alcoholic fermentation; and the removal of the dregs and scum, which form in considerable quantity, by racking as often as may be necessary, supplemented in some cases by filtration of the liquid. Sometimes catechu or other tannin matter is employed to precipitate albuminous matters, occasionally anti-acids are used to correct undue acidity, and preservatives, especially salicylic and boric acids, are stated to be frequently employed. An extensively-advertised preservative of cider, which I purchased in Bristol, was found on analysis to consist of calcium sulphite. According to Mr. G. Embrey, in Gloucestershire, an addition of 1 quart of milk to 18 gallons of cider is often made for fining the liquid, and as a preservative salicylic acid is frequently used in a proportion of 1 ounce to 96 gallons.

The apples suitable for the manufacture of cider are in many cases grown especially for the purpose, and are somewhat unpalatable, as they contain considerably more tannin than ordinary table apples. Ordinary table apples are unsuited for the production of cider, and, in fact, but few varieties of apple can be advantageously used alone, the practice of skilled cider-makers being to employ a mixture of different kinds of apple, so that the excess of certain constituents in some kinds may compensate for the deficiency in others.

Among the most esteemed cider-apples of the South-west and West of England are the Fox Whelp, Royal Wilding, Sweet Alfred, White Normandy Beech, Tom Put, White and Yellow Styres, Handsome Mandy, Kingstone Black, and Skyrme's Kernel.

In America, cider is made from the Baldwin, Newtown Pippin, Sweet Vandever, Rosebery Russet, Rambo, and many other varieties of apple, some of which are ill-suited for the purpose.

There is no doubt that in some parts of England the apple-trees have been so neglected that their fruit has degenerated, and contains an abnormal proportion of acid. The trees in some districts are rarely, if ever, pruned or manured, and the apples—windfalls, ripe and rotten fruit together—are allowed to remain in the orchard for many weeks, in some cases not even being stacked or protected from pollution by animal excreta. Starting with a mixture of unripe and more or less damaged fruit, with possible admixture of foreign matters, it is not surprising that the cider made and consumed by the rural population of the cider districts is apt to be of very inferior quality. The manufacturers of cider for public sale, of course, proceed in a more systematic manner, but they are largely dependent on the defective fruit described above. Cider is, however, now manufactured in England in increasing quantities under conditions in which the above defects are carefully guarded against, and a product of unexceptionable quality results.

Opinions as to the effect of apple-pips on the quality of cider vary greatly. Some cider manufacturers aim at crushing them, while others as carefully avoid doing so.

It is not generally recognised that under the generic term "cider" are grouped a number of products of the fermentation of apple-juice, which products vary among themselves almost as greatly as the numerous products from the fermentation of grape-juice grouped together under the generic name of "wine." Yet no distinctive names are given to the different classes of cider, except such as "sweet," "dry," "sparkling," etc. The geographical origin allows of some differentiation in the character of cider, but the terms "Devonshire," "Herefordshire," "American," etc., have not the same definite meaning that attach to the names Burgundy, Bordeaux, Champagne, Port, etc., in the case of wine. The proportion of sugar varies in cider from 10 to less than 1 per cent., diminishing with the age, while the proportion of alcohol increases. A serious practical difficulty in the classification of cider is its tendency to undergo both alcoholic and acetic fermentation in addition to more obscure changes, so that it is very difficult to insure that any particular brand shall be of approximately constant character.

In the case of sweet cider, which is necessarily only partly fermented, the proportion of alcohol is generally too low to preserve the liquid from further change, and hence a foreign antiseptic agent, such as salicylic or boric acid, is sometimes added. A "dry" cider, in which more perfect fermentation has occurred, is to be preferred on several grounds, but the manufacture of a natural product of this kind presents considerable difficulties, and the demand for it is limited. As long as the cider contains a fair proportion of sugar the sweet taste masks the presence of any excess of acid, but in dry cider this becomes unpleasantly apparent, unless some supplementary treatment be adopted.

The following table shows the chief constituents of *apples* analysed in my laboratory:

TABLE I.

	TABLE APPLE.	COOKING APPLE.	CIDER APPLES.	
	Blenheim Orange.	Derby.	Sweet Alfred.	Tom Put.
Water	81·62	84·74	80·29	84·14
Free acid (as malic) ...	0·88	0·56	0·097	0·36
Glucose...	9·28	8·75	9·43	7·21
Sucrose...	6·28	2·29	2·95	2·84
Ash	0·44	0·33	0·54	0·44

Many published analyses of apples and apple-juice are misleading, owing to the observers having ignored the presence of non-reducing sugars. Such sugar (presumably sucrose) is always present, and sometimes in considerable proportion. When the fruit is over-ripe the proportion of sucrose diminishes, a portion of it undergoing inversion, though part apparently undergoes a less simple change. The following figures, obtained in my laboratory, show the change resulting from decay in the case of two typical Devonshire cider-apples. The decayed portions were carefully separated from the still sound parts of the fruits, and each fraction analysed separately.

	No. 1 Apple.		No. 2 Apple.	
	Sound Part.	Decayed Part.	Sound Part.	Decayed Part.
Moisture	80·50	81·68	—	—
Volatile acid	0·016	0·057	—	—
Fixed acid	0·109	0·436	—	—
Glucose (reducing sugar) ...	9·30	11·86	9·49	11·61
Sucrose (non-reducing sugar) ...	6·67	1·49	4·74	0·98

The composition of the pressed apple-pulp, or *marc*, obtained in large Devonshire cider factories is shown by the following figures. In the cases of the first three samples the apples had been pressed once only, but in the case of the fourth the pulp had been broken up and re-pressed:

	1	2	3	4
Moisture	68·14	68·14	75·62	63·36
Glucose	4·04	—	4·69	2·24
Sucrose	2·14	—	2·26	1·88
Ash	—	1·01	1·03	1·71

The large proportion of water in apple-marc renders any useful application of the product very difficult. To a limited extent, and when quite fresh, it is sometimes employed as a cattle food, in other instances it is burnt under boilers fitted with a special contrivance, and in other cases it is taken out to sea.

The following table shows the variation (grammes per 100 c.c.) in the chief constituents of *apple* and *pear juice* from various parts of France, as recorded by Truelle:

TABLE II.

	Apple Must.			Pear Must.		
	Minimum.	Maximum.	Average.	Minimum.	Maximum.	Average.
Specific gravity ...	1057·5	1110·0	1076·0	1067·5	1098·0	1084·5
Total solids calculated from specific gravity	14·94	28·57	19·74	17·53	25·32	21·95
Acidity (as H_2SO_4) ...	0·07	0·74	0·21	0·08	0·24	0·15
Sucrose	0·56	7·17	2·50	1·67	6·14	3·67
Glucose	10·84	18·18	13·58	10·81	20·99	14·56
Pectous and albuminous matters ...	—	2·3	1·2	0·3	1·8	1·31
Tannin	0·026	0·81	0·29	0·10	0·32	0·18

The following results show the composition of fresh *apple-juice* as used for the manufacture of cider in the South-west of England. The formation of alcohol occurs very rapidly, and hence a notable proportion was found in several cases, though the analysis was commenced within thirty-six hours of the apples being pressed.

TABLE III.

	A.	B.	C.	D.	E.
Specific gravity	1·0550	1·0530	—	1·0470	1·0470
Alcohol, present by weight ...	0·10	—	—	1·04	1·13
Total extractive matters ...	14·63	12·74	16·11	11·91	11·95
Glucose	(13·51)	10·48	12·50	9·13	8·82
Sucrose...	1·34	0·69	—	0·66	0·38
Fixed acid (as malic)... ...	0·28	0·42	0·12	0·45	0·50
Ash	0·35	0·30	—	0·22	0·26
Tannin	—	0·22	0·11	—	—
Original solids...	14·84	12·74	16·11	14·05	14·28

The next three tables show the results yielded by the analysis in my laboratory of typical English ciders.

TABLE IV.

Analysis of Six Varieties of Norfolk Bottled Cider.

		A.	B.	C.	D.	E.	F.
Specific gravity ...		1·005	1·002	1·012	1·012	1·012	1·002
Alcohol, by weight ...		5·30	7·21	6·56	7·29	7·14	7·69
= Proof spirit ...		11·60	15·72	14·34	15·88	15·57	16·73
Grammes per 100 c.c.	Total extractive matters ...	2·07	2·54	4·71	5·47	4·55	2·33
	Glucose ...	0·77	1·16	4·16	4·55	3·12	0·88
	Fixed acid (as malic) ...	0·35	0·42	0·39	0·42	0·41	0·31
	Volatile acid (as acetic) ...	0·07	0·21	0·15	0·11	0·10	0·10
	Ash	0·30	0·26	0·31	0·33	0·27	0·26
Original solids ...		13·09	17·71	18·45	20·65	19·41	18·32

TABLE V.

Analysis of Typical Samples of Devonshire Bottled Cider.

	G.	H.	I.	J.	K.	L.	M.*	N.*	O.	P.	Q.
Specific gravity	1·012	1·003	1·013	1·006	1·032	1·011	1·029	1·0275	1·0230	1·0260	1·0190
Alcohol, per cent. by weight ...	4·77	5·39	5·09	4·95	2·57	4·09	4·01	3·77	4·18	3·43	4·62
= Proof spirit	10·44	11·79	11·14	10·82	5·63	8·96	8·79	8·28	9·16	7·53	10·13
Total extractive matters	4·09	2·80	4·27	2·12	8·23	3·88	7·77	7·93	7·13	7·73	6·25
Glucose ...	2·94	1·43	2·27	0·94	7·24	2·17	6·76	6·85	5·62	5·68	4·90
Fixed acid (as malic) ...	0·15	0·19	0·33	0·12	0·31	0·21	0·30	0·33	0·35	0·34	0·22
Volatile acid (as acetic) ...	0·19	0·32	0·24	0·20	0·37	0·31	0·30	0·23	0·24	0·20	0·28
Ash	0·23	0·29	0·34	0·25	0·34	0·30	0·27	0·25	0·31	0·41	0·36
Original solids	14·20	14·39	15·12	12·62	14·08	12·77	16·48	16·05	16·10	15·10	16·19
Total sugar (as glucose) ...	—	—	—	—	—	—	—	—	6·25	6·76	5·10

* A small quantity of sugar solution was added to these ciders.

TABLE VI.

Analysis of English Draught Ciders of Good Quality.

		R.	S.	T.	U.	V.	W.
Specific gravity ...		—	1·012	1·027	1·016	1·006	1·028
Alcohol, per cent. by weight		5·86	3·96	3·86	4·06	4·37	2·49
= Proof spirit ...		12·83	8·68	8·47	8·90	9·58	5·47
Grammes per 100 c.c.	Total extractive matters ...	7·63	3·04	8·14	5·18	2·59	7·52
	Glucose ...	—	—	4·17	2·94	0·93	6·17
	Sucrose ...						
	Fixed acid (as malic) ...	0·41	0·43	0·26	0·31	0·25	0·20
	Volatile acid (as acetic) ...	0·21	0·18	0·16	0·18	0·20	0·23
	Ash	0·22	0·22	0·34	0·24	0·35	0·24
Original solids ...		20·01	11·47	16·39	13·81	11·89	12·99

The following is an epitome of an elaborate research on the composition of French ciders by Eugene Grignon (*Le Cidre*, Paris, 1887). The figures for alcohol are given in c.c. per 100 c.c. of cider, but the other figures (given originally in grammes per litre) are expressed in grammes per 100 c.c. of the cider. The samples in Columns A and C were made from pure apple-juice, without any addition of water. Samples in Columns B and D were ciders to which known proportions of water had been added, and were consumed in the districts in which they were produced; but the figures in the table are corrected for the water added, and so represent undiluted ciders. Column E shows the composition of "renourris" ciders from the Department of the Eure. These are ciders five or six years old, which have received annually an addition of must sufficient to make up for the loss by evaporation. They are relatively rich in alcohol and low in extractive matters, the pectous bodies undergoing gradual precipitation.

To the actual results of analysis, Grignon appends the calculated amounts of alcohol and extractive matter which would be found in the cider if it had undergone complete fermentation. This is effected by deducting 0·1 per cent. from the sugar found by analysis (as an allowance for matters other than sugar which reduce alkaline copper solution, and are not removed by a preliminary treatment with basic lead acetate), and multiplying the difference by 0·6117, on the ground that 100 grammes of invert sugar yield on fermentation 61·17 c.c. (=48·55 grammes) of alcohol. Thus, if a sweet cider contain 4·0 per cent. of alcohol (by volume) with 6·64 grammes per 100 c.c. of total extract, including 3·57 of sugar, then the completely fermented liquid will contain 6·12 c.c. of alcohol and 3·17 grammes of extract per 100 c.c.*

An additional allowance might advantageously be made for the acetic acid present in sour cider. Each gramme of this corresponds very nearly to 1 c.c. of alcohol.

The free acid of the cider is stated by Grignon in terms of sulphuric acid, instead of the fixed acid being calculated as malic and the volatile acid as acetic acid. One c.c. of normal alkali will neutralize 49 milligrammes of H_2SO_4, 60 of $HC_2H_3O_2$, or 67 of $H_2C_4H_4O_6$, so that no large error results from the fact that the fixed and volatile acids are not differentiated. In the following tables I have added to Grignon's figures a statement of the original solids of the juice, but in calculating them I have made a constant allowance of 0·2 per cent. for sugar lost from the extract through acetic fermentation.

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* 3·57 of sugar found, minus 0·10, leaves 3·47 of corrected sugar in sample. 6·64 of extract, minus 3·47, leaves 3·17 extract in the completely-fermented cider. 3·47 of corrected sugar, multiplied by 0·6117, gives 2·12 increase in alcohol, which added to 4·00 in original sample, gives 6·12 c.c. of alcohol per 100 c.c. of completely-fermented cider.

TABLE VII.

Analyses of French Ciders (E. Grignon).

	A. Sweet Sparkling.	B. Sweet.	C. Dry.	D. Dry.	E. Renourri.
Number of samples contributing to average	5	4	30	15	10
Alcohol (by volume)	3·8	4·1	5·4	5·4	7·0
Extract (dried at 100° C.)	6·41	6·40	3·03	2·95	2·22
Ash	0·29	0·28	0·27	0·26	0·24
Acidity (as H_2SO_4)	0·36	0·39	0·52	0·58	0·54
Sugar	3·47	3·75	0·65	0·58	0·27
Polarimetric indication	-1°11′	—	-0° 12′	-0° 04′	-0° 02′
Alcohol after complete fermentation	5·9	6·3	5·8	5·7	7·1
Extract " " "	3·04	2·74	2·48	2·47	2·05
Original solids of juice (Allen) ...	12·77	13·24	11·97	11·90	13·76

The following figures show the extreme and mean composition of the sixty-four *ciders* included in the above table:

TABLE VIII.

	Minimum.	Maximum.	Average.
Alcohol (by volume) after complete fermentation	3·8	8·0	6·0
Extract, after complete fermentation	1·71	4·50	2·47
Ash	0·17	0·35	0·27
Acidity (as H_2SO_4)	0·27	0·71	0·52
Original solids of juice	—	—	12·39

The sample containing only 1·71 of extract was a *renourri* cider. In the greater number of cases the extract was considerably higher. The ash was usually notably higher than 0·17, the sample yielding this exceptionally small proportion showing 6·3 of alcohol (calculated) after complete fermentation.

The following figures show the average composition of *pure ciders* from various parts of France analysed in the Paris Municipal Laboratory:

TABLE IX.

	Well-fermented Cider.	Sweet Cider.
Alcohol, per cent. by volume	5·2	1·7
Solids, grammes per 100 c.c.	4·12	6·69
Sugar " "	0·89	2·91
Ash " "	0·29	0·26

From these results the Paris authorities ruled that pure, well-fermented ciders of the ordinary type should contain :

	Average.	Minimum.
Alcohol, per cent. by volume	5 to 6	3·0
Extract, grammes per 100 c.c.	3·0	1·8
Ash ,, ,, ...	0·28	0·17

Analyses of ciders from various parts of France, by G. Lechartier (*Compt. rend.*, ciii., 1104) show considerable departure from the foregoing figures.

It must be borne in mind that low alcoholic strength implies a high extract, so that the figures representing the minimum limits will never be met with in the same cider.

Perry presents a very close general resemblance to cider, but has a sweeter taste. This is not due so much to a larger proportion of sugar as to the smaller proportion of malic acid, the presence of which in cider masks the taste of the sugar. Pears usually contain very little acid, but are often rich in tannin and mucilaginous matters; and hence the fermented product is more difficult to deal with than one prepared from a mixture of apples with pears called in France *cidresse*. Pears intended for the production of perry should be aromatic, rich in sugar and tannin, moderately acid, and at the exact point of ripeness. A large portion of the perry produced in France is distilled for the production of spirit.

The following analyses of *sparkling perry* from Worcestershire and Devonshire were made in my laboratory. The analysis of Gloucestershire perry is by Mr. G. Embrey:

TABLE X.

	Worcestershire.	Devonshire.	Gloucestershire.
Specific gravity	1020·0	1021·0	1010·0
Alcohol, per cent. by weight ...	4·61	4·81	3·64
= Proof spirit per cent.	10·11	10·54	7·98
Total solids	6·51	6·49	4·50
Volatile acid (as acetic)	0·41	0·35	0·22
Fixed acid (as malic)	0·25	0·20	0·24
Glucose	2·71	3·60	0·36
Sucrose	none	0·31	—
Ash	0·40	0·28	0·30
Original solids	16·61	16·92	12·33

With regard to the sophistications of cider, by far the most usual is dilution with water either before or after fermentation. How far it is legitimate to treat the once-pressed marc with water, and, after again pressing, to add the washing so obtained to the pure juice, is open to question.

Tartaric and citric acid are sometimes added to cure diseases of cider, and in other instances preservatives are used.

With regard to preservatives, I may say that in English ciders made by large manufacturers I have not met with any salicylic acid, though it is said to be commonly used in cider of American origin. Another preservative said to be largely employed by cider manufacturers is boric acid. At the commencement of the investigation on which this paper is founded I found boric acid in every sample of cider I examined, and naturally supposed its use as a preservative to be very general; but an examination of the apples themselves showed that boric acid was a constituent of apples to such an extent as to be very misleading, if the fact of its natural presence were ignored. As it is, a very strong reaction with turmeric is obtainable from 20 grammes of cider, apple-juice, or from the fruit itself. Of course, boric acid has been recognised as a natural constituent in minute quantity in various plants. Thus, it has been found in all parts of the vine and in hops, but not in malt or barley. Gassend found it in grapes, apples, and in some pears, but not in all; and he states that it is absent from tea, saffron, and cows' milk. Hodder found it in fruits generally, Crampton denies its presence in apples or cider, while Jay states that cider and perry contain from 0·011 to 0·017 gramme per litre.

A considerable number of experiments on the presence of boric acid in apples and other fruit have been made in my laboratory by Mr. A. R. Tankard, who found it in all the apples examined, and also in pears, quinces, pomegranates and grapes. In a specimen of "Fox Whelp" apple the amount of boric acid was 0·0076 per cent.; in quince half as much again. In pears the proportion appears to be variable.

Considerable trouble has been taken to arrive at a satisfactory method of determining these small quantities of boric acid in the presence of excess of phosphates, etc., and a satisfactory process was only devised after a large number of experiments had been made. The process actually employed must form the subject of a subsequent paper.

Dilution of cider will generally be indicated by deficiency in the proportions of alcohol and extractive matters. This can be calculated by the method of Grignon already described, but an alternative and preferable plan is to calculate the original solids of the juice from the results of the analysis, thus:

Alcohol, per cent. by weight × 2·07 = sugar fermented ...
Acetic acid found × 1·5 = sugar fermented ... ...
Extractive matter in sample ... ...

Sum = original solids of juice ... ...

The original solids of unwatered cider when thus calculated rarely fall below 12 per cent. On the other hand, an excessive proportion of original solids points to an addition of saccharine matter.

A valuable corroboration of the deduction from the foregoing method is obtained by a determination of the ash of the cider. According to G. Embrey, this should range from 0·25 to 0·35 per cent., and should not fall below the former figure. In American ciders a larger proportion of ash is usually met with, but for French ciders the limit of 0·25 per cent. would be too high. The addition of mineral matters such as alkaline carbonates, lime, or boric acid would tend to increase the ash, and thereby invalidate the inference.

In 1895, as a result of a careful investigation and consultation with the Pomological Society of France, the Bath and West and Southern Counties Society recommended that no cider should be considered genuine which did not contain at least 4 per cent. of alcohol by volume (=7 per cent. of proof spirit). This provision is insufficient unless supplemented by a requirement as to sugar or extractive matter. Incompletely fermented (sweet) cider of excellent quality might readily fail to comply with the foregoing requirement,* although by fermentation of the residual sugar the matured product would contain nearer 6 than 4 per cent. of alcohol.

I have to acknowledge the valuable assistance received from Mr. Jas. Thompson and Mr. A. R. Tankard in the execution of the greater number of the numerous analyses made in connection with the enquiry.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

Rapid Detection of Formaldehyde in Articles of Food. C. Arnold and C. Mentzel. (*Chem. Zeit.*, 1902, xxvi., 246.)—A little phenylhydrazine hydrochloride is dissolved in 3 to 5 c.c. of the cold liquid which is to be tested, and 2 to 4 drops (not more) of a 5 to 10 per cent. solution of sodium nitroprusside are added; then 8 to 12 drops of 10 to 15 per cent. alkali hydroxide are run in one at a time, when, according to the amount of formaldehyde present, a blue or blue-gray colour appears immediately, which is permanent for some time. In the case of fats, 5 to 10 grammes are shaken at the melting-point with 10 c.c. of 80 to 95 per cent. alcohol, cooled, the solution filtered through a moist paper, and examined as before. Raw or boiled milk, containing 0·015 gramme of formaldehyde per litre, gives a distinct green; pure milk is only tinted yellow. 0·05 gramme per litre yields a fine blue. Raw chopped flesh, saturated with a 0·5 per cent. solution of formaldehyde, was extracted with double its volume of hot water; the expressed and filtered liquor gave the reaction well. It also succeeded after keeping the meat for a day. With fats the test indicates 0·3 gramme per kilo.

A still more delicate reaction is given by substituting potassium ferricyanide for the nitroprusside; this develops an intense scarlet in presence of formaldehyde, permanent for a day. Unlike the former test, this only succeeds in alcoholic liquids when enough water to hold the ferricyanide in solution is added. The second reaction is not to be recommended for the examination of milk; on flesh it is only to be used if the material is not reddened by blood-pigments. In all other cases it may be employed if the first test fails by excessive dilution. F. H. L.

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* An excellent cider made by one of the leading manufacturers was disqualified some years since on this insufficient ground.

The Detection of Aloes in Pharmaceutical Preparations. E. Léger. (*Journ. Pharm. Chim.*, 1902, xv., 335.)—*Identification of the Type of Aloes.*—In order to identify aloes and to distinguish between those of the type of Cape aloes and those of Barbadoes or Curaçoa, the author makes use of Klunge's reaction in the following manner: A solution of 0·5 gramme of aloes in 100 c.c. of hot water is rapidly cooled in a current of cold water and filtered from the deposited resin, the filtration being accelerated by the addition of a little powdered talc.

The following tests are then applied to the clear yellow filtrate:

1. A few decigrammes of sodium peroxide are introduced little by little into 20 c.c. of the liquid, previously heated to 80° C. An immediate evolution of oxygen occurs, and the liquid becomes brown, subsequently changing to bright cherry-red on the further addition of peroxide. The author has identified various substances in the products of the reaction, but attributes the red coloration to the presence of the alo-emodin of Tschirch and Oesterle (*Ber. d. d. Pharm. Ges.*, viii., 174; *Archiv. der Pharm.*, ccxxxvii., 81).

2. A second portion of 20 c.c. of the filtrate is treated with 1 drop of a saturated solution of copper sulphate, which causes the yellow colour to become darker. One gramme of pure crystallized sodium chloride is next introduced, and then 10 c.c. of 90 per cent. alcohol, which dissolves the flocculent precipitate produced by the salt.

With Socotrine and Cape aloes a vinous red colour is obtained, which gradually fades, until after an hour the liquid has become yellow, and shows no further change. In the case of Barbados and Curaçoa aloes, however, a bright, currant-red coloration is obtained, which does not disappear even after standing for twelve hours. The author has found that this reaction is due to a special aloin, which he terms barbaloin, in the latter varieties of aloes.

Both of the above reactions are very sensitive, the first being obtained with solutions containing only 0·1 per cent. of aloes. Where the coloration is faint, the liquid should be acidified with hydrochloric acid and extracted with ether, and the ethereal solution of emodin shaken with 2 to 3 c.c. of water, rendered alkaline with sodium hydroxide, when a bright red colour will be obtained.

Detection of Aloes.—For the detection of aloes in pharmaceutical preparations the first (sodium peroxide) reaction is recommended. It frequently happens, however, that other drugs are also present, such as rhubarb, which contains chrysophanic acid (dioxymethylanthraquinone), an emodine (trioxymethylanthraquinone), and rheine (tetraoxymethylanthraquinone), which dissolve in sufficient quantity to falsify the results. The presence of oxymethylanthraquinones can be detected by adding a small quantity of sodium hydroxide solution, with which they give an immediate red coloration, whilst the solution of pure aloes remains yellow.

If the red colour be obtained, a slight excess of subacetate of lead is added to the solution to precipitate the oxymethylanthraquinones; or the following method of separation may be employed: Fifty c.c. of the filtered solution of 1 gramme of aloes in 100 c.c. of water are treated with 20 c.c. of a 5 per cent. solution of alum, then with ammonium hydroxide in excess, and lastly with acetic acid until the liquid is just acid. Twenty c.c. of the clear filtrate tested with sodium peroxide gives the aloes reaction clearly, whilst rhubarb extract treated in the same way gives only a faint peach coloration.

As a general rule, however, the author recommends the precipitation with subacetate of lead, which he shows gives good results with mixtures of aloes with *Cascara sagrada* and with *Rhamnus frangula*. C. A. M.

On the Reactions for the Identification of Certain Drugs. E. Bourquelot. (*Journ. Pharm. Chim.*, 1902, xv., 342-345.)—*Aloes.*—Klunge's reaction (see preceding abstract) having been shown to be one of oxidation, the author has made experiments to determine whether the same red coloration could be obtained by the action of oxidizing enzymes. For this purpose he used a 50 per cent. glycerin extract of *Russula delica*, and obtained a pronounced purple-red colour on adding 10 to 20 drops of this extract to 20 c.c. of a solution of aloin (1 : 500). As in the case of Klunge's reaction, the intensity of the coloration obtained diminished on recrystallization of the aloin, until eventually there was no reaction, owing to the elimination of the barbaloin.

Tyrosine.—Hirschsohn's reagents for aloin (copper sulphate and hydrogen peroxide) also react with tyrosine, a brown coloration being obtained on heating the solution with 1 drop of a 1 per cent. solution of copper sulphate and 2 drops of hydrogen peroxide solution.

Loganine—the glucoside discovered by Dunstan and Short—is present in tinctures of nux vomica and St. Ignatius' bean. The residue left on evaporating a few drops of the tincture in a porcelain basin, and warmed with 3 drops of dilute sulphuric acid (1 : 3), assumes a reddish-violet colour.

Preparations of Opium.—In order to identify the presence of meconic acid, 2 c.c. of the tincture are mixed with 4 c.c. of water and a few drops of hydrochloric acid, the mixture shaken with ether, and the ethereal extract shaken with 2 to 3 c.c. of water and a drop of ferric chloride, a red colour being produced in the aqueous layer.

Tincture of Cachou.—In the new German Pharmacopœia this is stated to give a deep red colour when heated with potassium chromate solution. As the tincture itself is very dark, the author modifies the test by boiling together 20 c.c. of water, 10 drops of the tincture, and 5 drops of potassium chromate solution (1 : 20), a cherry red colour being immediately produced. C. A. M.

TOXICOLOGICAL ANALYSIS.

Differentiation of Human Blood from the Blood of Animals. De Nobele. (*Ann. Soc. Med. de Ghent*, 1901, 331; through *Ann. de Chim. anal.*, 1902, vii., 150-152.)—The author has studied the method devised by Wassermann and Schütze, who found that the serum of a rabbit or a mouse into which human serum had been injected gave a precipitate when mixed with the serum of human blood.

He has proved that the injection of different human excreted liquids, such as the sera of milk, saliva, pus, albuminous urine, or ascitic fluid, has also the same effect upon the serum of the animal, which in each case gives a precipitate with human blood, but not with the blood of different animals.

Reactions were also obtained with decomposed human blood. Thus, stains on linen from several days to two months in age, when treated with very dilute sodium chloride solution or with sodium hydroxide solution (0·1 per cent.), gave solutions yielding a precipitate with the injected serum. No reaction was given, however, by a stain nine years old.

To determine the action of heat, drops of blood were heated on glass to temperatures of 75°, 100°, and 125° C., and the dried residues dissolved in sodium chloride solution and tested. Reactions were obtained with the blood dried at the lower temperatures, but not with that dried at 125° C.

Attempts to prepare a specific serum by feeding the animal with human serum instead of by inoculation were unsuccessful.

Corin (*Ann. Soc. Med. legal. Belg.*, i. [13]) has found that the active agent in the injected serum is a paraglobulin, which can be precipitated with magnesium sulphate. The precipitate, collected on a filter and dried, can be kept unaltered, and when required can be redissolved.

According to the author, the serum can also be evaporated under reduced pressure without losing its activity. The dried residue is preserved in sealed tubes in the dark, and has been found to react as strongly as the original liquid after six months (*cf.* ANALYST, xxvii., 157). C. A. M.

Comparison of the Stas-Otto and Kippenberger Methods of detecting Alkaloids in Toxicology. J. Weiss. (*Münch. medicin. Wochenschr.*, 1902, xlix., 367; through *Chem. Zeit. Rep.*, 1902, 100.)—The author has carried out experiments on the detection of strychnine, morphine, and atropine in nutritive liquors, in the stomachs and intestines of corpses, and in whole corpses, soon after death and also a considerable time after burial. In all cases the yield of alkaloid was greater by the Stas-Otto than by the Kippenberger process. It is therefore better to employ the former for the isolation of the substance, and only to use Kippenberger's method with iodine and potassium iodide for quantitative purposes. F. H. L.

ORGANIC ANALYSIS.

Colour Reaction for Wood-Fibre. A. Kaiser. (*Chem. Zeit.*, 1902, xxvi., 335.)—Equal volumes of amyl alcohol, free from furfural, and strong sulphuric acid are warmed on the water-bath to about 90° C., until a slight evolution of gas occurs; the faintly orange mixture is then cooled. This reagent, which may be termed "amyl-sulphuric acid," has the property of turning wood-fibre red, violet, or dark indigo-blue, according to the quantity present. Newspaper or pine shavings moistened therewith strike first a greenish, afterwards a bright blue colour; while pure Swedish filter-paper only becomes red and commoner qualities violet. Appearance of the tint is hastened by a current of air and by gentle warming. As the colours vary with the quantity, it is possible roughly to gauge the proportion of wood-fibre present in a sample of paper. The whole process is an adaptation of Vitali's reaction.

F. H. L.

The Alkalinity of Crude Sugar. Herberger. (*Centralbl. Zuckerind.*, 1902, x., 552; through *Chem. Zeit. Rep.*, 1902, 92.)—The author states that phenolphthalein is useless for determining the alkalinity of crude sugar, since various salts and organic substances are present in the material which cause it to appear acid when it is really alkaline. F. H. L.

Quantitative Estimation of Arabinose. C. Neuberg and J. Wohlgemuth. (*Zeits. physiol. Chem.*, 1902, xxxv., 31; through *Chem. Zeit. Rep.*, 1902, 108.)—In the course of a research in which large quantities of the three arabinoses and their derivatives were required, the authors tested the following method. One hundred c.c. of urine, to which 1 gramme of arabinose had been added, was acidified with two drops of 30 per cent. acetic acid, concentrated on the water-bath to 40 c.c., and treated with 40 c.c. of hot 96 per cent. alcohol. After cooling for two hours the deposited salts were filtered off and washed with 40 c.c. of 50 per cent. spirit. The filtrate was mixed with 1·4 grammes of diphenylhydrazine, warmed in a boiling water-bath for half an hour, allowed to stand for twenty-four hours, and then filtered through a Gooch crucible, using the liquor for rinsing purposes. The crystals were then washed with 30 c.c. of 30 per cent. alcohol, which left them brilliantly white. The crucible was heated in the drying oven at 80° C. to constant weight, when the hydrazone assumed a faint violet tint, and the yield was found to be 2·1055 grammes of hydrazone, corresponding with 0·9993 gramme of *l*-arabinose, or 99·93 per cent. of the theoretical. Similar tests with *d*-arabinose gave a return of 99·80 per cent., and with *i*-arabinose 100·06 per cent. The method is therefore available for the separation of this sugar from other carbohydrates, especially xylose, even when the latter is present in considerable excess. It is necessary that 1 per cent. of arabinose be present; weaker liquids must be previously concentrated *in vacuo*. F. H. L.

On Stahre's Reaction for Citric Acid. A. Wöhlk. (*Zeit. anal. Chem.*, 1902, xli., 77-100.)—The sensitive reaction for citric acid discovered by Stahre (*Nordisk Farm. Tidsskrift*, 1895, ii., 141) is based upon the fact that on oxidizing citric acid with potassium permanganate and adding bromine a white precipitate insoluble in ether is obtained. Stahre concluded that in this reaction the citric acid was oxidized to acetone, which was converted by the bromine into a bromacetone.

In applying the test when less than 5 milligrammes of citric acid are present, the acid is dissolved in 2 c.c. of water, and heated to 30° C. after the addition of 2 to 4 drops of $\frac{N}{10}$ permanganate solution. If any slight precipitation of manganese peroxide occur, 1 or 2 drops of a 4 per cent. solution of ammonium oxalate and about 1 c.c. of 10 per cent. sulphuric acid are introduced to clear the liquid. On now adding 2 drops of bromine water a distinct turbidity is produced.

When more than 5 milligrammes of citric acid are present the solution in 2 c.c. of water is treated with 5 drops of the permanganate solution, heated to 30° to 40° C., cooled, and mixed with 1 c.c. of dilute sulphuric acid. Should any precipitate be formed it can be removed by the addition of a little ammonium oxalate. Bromine water then added drop by drop gives a precipitate of extremely fine needles of pentabromacetone.

Stahre's reaction is obtained not only with pure solutions of citric acid and its salts, but also in the presence of other organic acids precipitated by calcium, such as tartaric, oxalic, malic acids, or inorganic acids, such as sulphuric and phosphoric acids. In such cases, however, more permanganate must be used, and it is best to make the test on a hot solution. Organic acids that react with bromine must obviously be removed.

The author confirms the statements of Henkel, Scheibe, and others as to the presence of citric acid as a normal constituent of milk. He has separated a compound which he has identified as citric acid by Stahre's reaction and other tests.

C. A. M.

The Determination of Mustard Oil. **P. Rœser.** (*Journ. Pharm. Chim.*, 1902, xv., 361-364.)—The method recommended by the author is based upon the conversion of the allyl isothiocyanate into thiosinamine (as in the methods of Gadamer, etc.), adding standard silver nitrate solution to the ammoniacal liquid, and titrating the excess of silver by Denigés' method.

Five c.c. of a 1 per cent. solution of the commercial essence are mixed with 10 c.c. of ammonium hydroxide, and, after dilution with water, shaken with 10 c.c. of $\frac{N}{10}$ silver nitrate solution. After standing for twenty-four hours, the liquid is made up to 100 c.c. and filtered, 50 c.c. of the filtrate treated with 5 c.c. $\frac{N}{10}$ potassium cyanide solution, and the excess of cyanide titrated with silver nitrate solution, a few drops of a slightly ammoniacal 5 per cent. solution of potassium iodide being used as indicator.

The number of c.c. of silver nitrate solution taken up by the sulphur, doubled and multiplied by the factor 0·3137, gives the percentage of mustard oil in the essence.

For the determination of mustard oil in mustard flour, leaves, etc., 5 grammes of the sample are mixed with 60 c.c. of water and 15 c.c. of 60 per cent. alcohol, and distilled after standing for two hours. The distillate is received in a flask containing 10 c.c. of ammonium hydroxide, and after two-thirds of the liquid have passed over, it is mixed with 10 c.c. of $\frac{N}{10}$ silver nitrate solution and diluted to 100 c.c., and the determination completed as described above.

It is stated that this method gives results almost identical with those obtained by the methods of Dieterich and Gadamer.

C. A. M.

Quantitative Separation of Cholesterin from Fats. E. Ritter. (*Zeits. physiol. Chem.*, 1902, xxxiv., 430; through *Chem. Zeit. Rep.*, 1902, 100.)—The author has submitted his own process (this vol., p. 16) and that proposed by Bömer to a comparison, using in each case a fat relatively rich in phytosterin. He finds that his own method returns rather more phytosterin than Bömer's process, and considers that many of the objections to the latter are avoided. The product is also purer, but it is not absolutely pure phytosterin. F. H. L.

The Composition of Cacao Butter. J. Klimont. (*Monatsh. für Chem.*, 1902, xxiii., 51-59.)—This paper is a continuation of the author's former communication on this subject (ANALYST, xxvii., 15), in which he stated that he had identified tripalmitin, tristearin, and a mixed glyceride containing the radicles of oleic, palmitic, and stearic acids, together with another mixed glyceride.

From a consideration of the elementary composition, iodine value, and saponification value of this last glyceride he has come to the conclusion that it contains the radicals of myristic, palmitic, and oleic acid, and possibly of some still lower fatty acids.

Neither arachidin nor arachidic acid could be detected in the specimen of fat examined. C. A. M.

A Note on the Use of the Bechi or Silver Nitrate Test on Olive Oils. L. M. Tolman. (*Journ. Amer. Chem. Soc.*, xxiv., 396.)—Since some olive oils free from cottonseed oil give a brown coloration with the Bechi test as generally applied, the author recommends the following preliminary treatment, which, whilst not affecting the reducing action of the cottonseed oil, removes the free acid and other products of rancidity of the olive oil: To 25 c.c. of the oil, 25 c.c. 95 per cent. alcohol are added, and the whole is gently heated and then vigorously shaken; after allowing the liquids to separate, the alcoholic solution is decanted off, and the residue washed first with 2 per cent. nitric acid and finally with water. For the test, 10 c.c. oil, 10 c.c. amyl alcohol, and 5 c.c. of the reagent (2 grammes silver nitrate dissolved in 200 c.c. alcohol, with the addition of 40 c.c. ether and 2 drops nitric acid), are mixed in a test-tube. Half the mixture is kept for comparison, the other half being heated for ten minutes in a boiling water-bath. The above method of purification is stated to give very satisfactory results, and can be readily applied to lard and other fats.

A. G. L.

Comparison of Methods used to Determine the Iodine Value of Oils. F. W. Hunt. (*Journ. Soc. Chem. Ind.*, 1902, xxi., 454.)—The methods compared were those of Hübl, Wijs, and Hanus (see ANALYST, xxvii., 15). He found that the Wijs solution (ICl in glacial acetic acid) lost 3 per cent. of its strength in two weeks, and the Hanus solution (IBr in glacial acetic acid) nearly 5 per cent. In determining the iodine values, Hübl's solution was allowed to act for five hours, the other two for one hour. Each determination was done in duplicate:

	Hübl.	Hanus.	Wijs.
Pale seal	117·5	120·7	124·8
Whale	120·2	120·6	123·7
Olive	82·3	81·9	83·4
Castor (cold drawn)	82·6	84·4	85·6
Dogfish liver	134·7	141·2	143·2
Arachis (earth-nut)	91·8	91·6	93·4
Arctic shark	89·0	93·8	98·9
Pale rape	103·0	101·9	102·1
Coast cod	148·5	147·5	154·6
Newfoundland cod	144·8	150·0	154·7
Linseed	174·8	174·5	177·3
Cottonseed	108·5	107·0	110·0

The Wijs solution gave results higher than the Hübl solution, especially in the case of fish oils. The Hanus solutions gave results which were in some cases intermediate and in others agreed with those given by Hübl's solution.

Experiments were also made using a solution of iodine monobromide in carbon tetrachloride. The results obtained were:

	Iodine Value.	Corrected for Substitution.
Olive oil	82·0	—
Coast oil	153·9	—
Linseed	179·7	177·8
Arachis	90·8	89·5
Pale seal	125·4	124·5

The author does not recommend this solution, however. A. M.

On the Temperature Reaction of Oils with Sulphuric Acid—Maumené's Test. H. C. Sherman, J. L. Danziger, and L. Kohnstamm. (*Journ. Amer. Chem. Soc.*, xxiv., 266.)—The authors have investigated the sources of errors in Maumené's test, especially as regards the drying oils, which give a violent reaction with the sulphuric acid, and come to the conclusion that if the oil is diluted with some non-drying oil the value calculated is always too high. They find that the use of a somewhat more dilute acid (89 to 90 per cent.) than is usually employed gives good results, which are directly comparable for both drying and non-drying oils. They recommend the statement of all results as "specific temperature reactions," according to the suggestion of Thomson and Ballantyne (*Journ. Soc. Chem. Ind.*, x., 233), which are obtained by multiplying the observed rise of temperature by 100, and dividing by the rise observed when 50 grammes water are treated with 10 c.c. of the same acid under the same conditions. Contrary to Thomson and Ballantyne, however, they find this value is slightly lower for a more dilute acid; but, still, their results agree reasonably well with those obtained by these authors. A. G. L.

Metallic Soaps from Linseed Oil. An Investigation of their Solubilities in Certain of the Hydrocarbons. Hermann T. Vulté and Harriet Winfield Gibson. (*Journ. Amer. Chem. Soc.*, xxiv., 215.)—The authors have investigated the solubilities of a number of metallic soaps from linseed oil in various petroleum solvents and in turpentine, as well as the permanence of the solutions, and give tables showing the results obtained. They find that the lead soap separates from its solutions in all the petroleum solvents in less than one hour, the nickel soap only after several days, the iron soap only on the application of a gentle heat, and that the manganese soap is unique as regards its drying properties They also find that no one solvent can be recommended for all the soaps, there being a maximum solubility for each one in some special solvent. A. G. L.

Characteristics of Butter from Human Milk. Sauvaitre. (*Ann. de Chim. nal.*, 1902, vii., 143-145.)—The author has obtained the following results with a specimen of human butter, and compares the figures with those given by a sample of cow's butter:

	Cow's Butter.	Human Butter.
Specific gravity at $\frac{100^\circ}{15^\circ}$ C.	0·866 per cent.	0·870 per cent.
Critical temperature of solution in absolute alcohol	56° C.	59° C.
Coefficient of solubility in absolute alcohol	43·3 per cent.	34·7 per cent.
Melting-point of insoluble fatty acids ...	40·5° C.	40° C.
Solidification-point ,, ,, ...	39° C.	37° C.
Saponification value	221·2 per cent.	218·4 per cent.
Reichert-Wollny value	26·3 ,,	15·8 ,,
Hehner value	87·2 ,,	89·2 ,,
Iodine value	35·51 ,,	43·37 ,,
Total volatile acids	6·69 ,,	4·41 ,,
Ratio of butyric acid to caproic acid ...	2·2 : 1	2·4 : 1
Unsaponifiable matter	1·58 per cent.	4·68 per cent.
Melting-point of unsaponifiable matter ...	139° C.	143° C.

C. A. M.

Detection of Indican in Urine. Bertault. (*Journ. Pharm. Chim.*, 1902, [6], xv., 277; through *Chem. Zeit. Rep.*, 1902, 92.)—If the urine contains iodine (or bromine) as well as indican, the well-known test must be modified as follows: After the sample has been treated with an equal volume of hydrochloric acid, a few c.c. of chloroform, and a few drops of sodium hypochlorite, the acidified urine is pipetted off, the tube containing the chloroform is washed with water to remove the bulk of the acid, and then potassium hydroxide is added to alkalinity. The violet colour produced by the iodine disappears, and the chloroform remains blue if indican is present, or colourless in its absence. F. H. L.

Valuation of Rubber Goods. A. Heintz. (*Chem. Zeit.*, 1902, xxvi., 247.)—The author suggests a method for the valuation of rubber goods, in which an attempt is made, by ultimate analysis, to determine the proportion of true caoutchouc present. After a qualitative examination, the various foreign organic substances are removed by treatment with Henriques' solvents: unsaponifiable oils by ether, asphaltum by nitrobenzene, "substitutes" by alcoholic soda. The last traces of these liquids are next most carefully washed away, the residue is thoroughly dried, a combustion is carried out, and the amount of caoutchouc is estimated from the gain of the sulphuric acid tube, on the assumption that the body remaining after the above process is represented by the formula $(C_{10}H_{16})_n$. The method is not offered as an exact process, but one for commercial purposes only. It suffers from two possible sources of error: (1) That the caoutchouc originally used in the manufactured goods does not correspond in composition with the formula quoted, but contains some oxygen compound, resins, etc.; (2) that the solvents do not remove the whole of, or remove substances other than, those they are supposed to extract. As regards the first point, analysis of the washed rubber employed in the manufacture of boots has shown that on an average it consists of 97 per cent. of $(C_{10}H_{16})_n$, so that even if the finished wares contained 50 per cent. of caoutchouc, which is unlikely, the error would be only 1·5 per cent. In order to settle the second point, analyses have been made of several trade samples of finished goods and of materials prepared specially for the author, both by his process and by that of Henriques, etc. Two samples of Russian-made shoes gave 57·63 and 60·43 per cent. of inorganic matter, 1·51 and 1·57 per cent. of sulphur, 19·23 and 18·62 per cent. of organic substance, or 21·63 and 19·38 per cent. of caoutchouc, by difference respectively. By the author's process they gave 20·23 and 18·65 per cent. of caoutchouc, or, after correction for the 3 per cent. of oxygenated matter, 20·84 and 19·21 per cent. A material composed of ceresin and brown "substitutes," beside the rubber and sulphur, gave 75·11 per cent. (corrected) on ultimate analysis, whereas it contained 76·75 per cent.* of caoutchouc. Another specimen containing inorganic matter, beside the sulphur and caoutchouc, gave 49·65 per cent. on combustion, the actual amount of rubber being 49·30 per cent.* F. H. L.

The Determination of Sulphur in Plants. G. S. Fraps. (*Journ. Amer. Chem. Soc.*, xxiv., 346.)—To 5 grammes of the material, placed in a 3½-inch porcelain dish, 20 c.c. concentrated nitric acid are gradually added; the mixture is heated cautiously on the water-bath, then concentrated, and, after the addition of 10 c.c. of a 5 per cent. solution of potassium nitrate, evaporated to dryness; the residue is ignited, at first gently, finally over the blast-lamp, and the sulphur in it estimated as usual.

A. G. L.

INORGANIC ANALYSIS.

The Electrolytic Determination of Copper in Iron. H. Koch. (*Zeit. anal. Chem.*, 1902, xli., 105-107.)—The copper is first concentrated by treating the iron with dilute sulphuric acid and then separated by electrolyzing the insoluble residue.

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* It is not clear whether by these figures the author means the yield of the Henriques process, or whether they are the proportion of caoutchouc actually employed by the manufacturer.—F. H. L.

In the case of steel, 100 grammes of the shavings are treated with 400 c.c. of dilute sulphuric acid (specific gravity, 1·06), half of this quantity being first used without the aid of heat, and the remainder added to the hot liquid after completion of the reaction. Crystallization of the sulphates during cooling is prevented by the addition of 500 c.c. of water.

The black residue is collected on a double filter, dried, ignited, and dissolved in a large porcelain crucible in a few c.c. of concentrated hydrochloric acid, and the solution evaporated to dryness after the addition of sulphuric acid. The residue is dissolved in about 20 c.c. of water and the same quantity of nitric acid (specific gravity 1·20), the solution filtered, and the filtrate diluted with water to about 120 c.c.

A few drops of a 5 per cent. solution of oxalic acid are then added, and the liquid electrolyzed for nine to ten hours with a current of about 0·094 ampère per square centimetre. The positive electrode consists of a spiral of platinum wire 1·3 millimetres thick, which rests upon the bottom of the beaker, whilst the negative electrode is a cylinder of platinum foil 9 by 3·5 centimetres in size.

The method requires a slight modification in the case of gray pig-iron, owing to the large proportion of graphitic carbon present protecting the iron from the action of the acid. To obviate this the residue should once more be boiled with about 200 c.c. of sulphuric acid (sp. gr. 1·06) for about thirty minutes before being treated as described above.

C. A. M.

Rapid Method for the Volumetric Determination of Molybdenum Steel. Francis T. Kopp. (*Journ. Amer. Chem. Soc.*, xxiv., 186.)—Half a gramme of the sample is dissolved in 2 c.c. sulphuric acid (specific gravity 1·58) and 12 c.c. water in a platinum crucible of 100 c.c. capacity, solution being hastened by heating. The solution is then evaporated until white fumes are given off, and cooled; to the residue 30 grammes fused potassium bisulphate are added, and the whole is then gently heated to a red heat, and kept at this temperature for ten or fifteen minutes. After cooling, the melt is dissolved in 500 c.c. hot water, the solution cooled, transferred to a litre flask, and, after the addition of 100 c.c. ammonia (specific gravity 0·90), made up to the mark with water. After mixing thoroughly 500 c.c. are filtered through a dry filter, 40 c.c. sulphuric acid (specific gravity 1·58) added, and the solution is run through a Jones reductor consisting of a column of zinc 12 inches long and 0·5 inch diameter. To the reduced solution 10 c.c. sulphuric acid are added, and the molybdenum is titrated with standard permanganate solution. A blank, consisting of 450 c.c. water, 50 c.c. ammonia, and 40 c.c. sulphuric acid, should be run through the reductor and titrated as above, in order to ascertain the error due to impurities in the zinc.

For tungsten steels, 1 gramme of the sample is dissolved in 25 c.c. dilute nitric acid, with the addition, after violent action is over, of 10 c.c. strong hydrochloric acid. The solution is evaporated to dryness, the residue baked, and then re-dissolved in 15 c.c. hydrochloric acid. The tungsten will be precipitated as tungsten trioxide. After cooling and diluting with water to 100 c.c., 50 c.c. of the solution are filtered through a dry filter, 10 c.c. of sulphuric acid are added, and the solution (which should be free from tungsten) is evaporated till white fumes are given off, transferred to a platinum crucible, again evaporated till white fumes are given off, cooled, and fused with 30 grammes potassium bisulphate, as in the case of steels.

For ferro-molybdenums 0·5 gramme of the sample is dissolved in 15 c.c. strong nitric acid in a platinum crucible; when thoroughly dissolved, 2 c.c. sulphuric acid are added, the solution is evaporated till white fumes are given off, and then treated as for steels.

The method gave good results when checked on steels containing known amounts of molybdenum, and also when compared with other methods of estimating molybdenum.

A. G. L.

Identification of Metals of the Platinum Group by means of Sodium Peroxide. Leidié and Quennessen. (*Bull. Soc. Chim.*, 1902, xxvii., 179-183.)—Various tests are commonly employed to differentiate the metals of platinum ores, and some of these are both tedious and also require fresh samples of the material to be taken.

The authors have made experiments to devise a rapid method in which all the tests can be applied to one and the same sample, and have eventually found that sodium peroxide at a dull red heat is a suitable reagent.

The metal, which should be in the finely divided state as obtained by ignition of the salt in hydrogen, is mixed with five or six times its weight of sodium peroxide, and gently heated in a nickel basin over a low gas flame. The mass first blackens and then becomes pasty, but after being stirred with a platinum spatula, and the heating continued, it becomes semi-liquid. The flame is now withdrawn, and the cold contents of the basin cautiously treated with water in the proportion of ten to twelve times the weight of sodium peroxide used. The mixture is now transferred to a tube, and, after standing, the clear liquid is decanted, and the deposit mixed with water and collected on a filter.

The oxide of nickel formed will be found in this deposit, and if insoluble oxides or insoluble alkaline salts of the platinum group are also present, it can be readily separated from them by means of the nitrite method previously described by Leidié (ANALYST, xxvi., 108).

The separate metals treated with sodium peroxide in this way give the following results:

Osmium is completely converted into sodium osmiate, $OsO_2(NaO)_2$, which dissolves in water, yielding a yellow solution. On passing a current of chlorine through this solution slightly heated, osmium peroxide, OsO_4, can be collected in a receiver immersed in ice water. The latter compound may be identified by the formation of potassium osmiate (action of potassium nitrite on heating, or of alcohol and potassium hydroxide in the cold).

Ruthenium is completely converted into sodium peruthenate, $RuO_3(NaO)$, which in the presence of water yields the ruthenate, $RuO_2(NaO)_2$. The latter forms an orange solution, which on treatment with chlorine gas, as above, yields a yellow liquid, in which ruthenium peroxide, RuO_4, may be identified by the reduction of ruthenium on treatment with potassium hydroxide and alcohol in the cold, or by the formation of a brown sesquichloride, Ru_2Cl_6, by the action of hot hydrochloric acid.

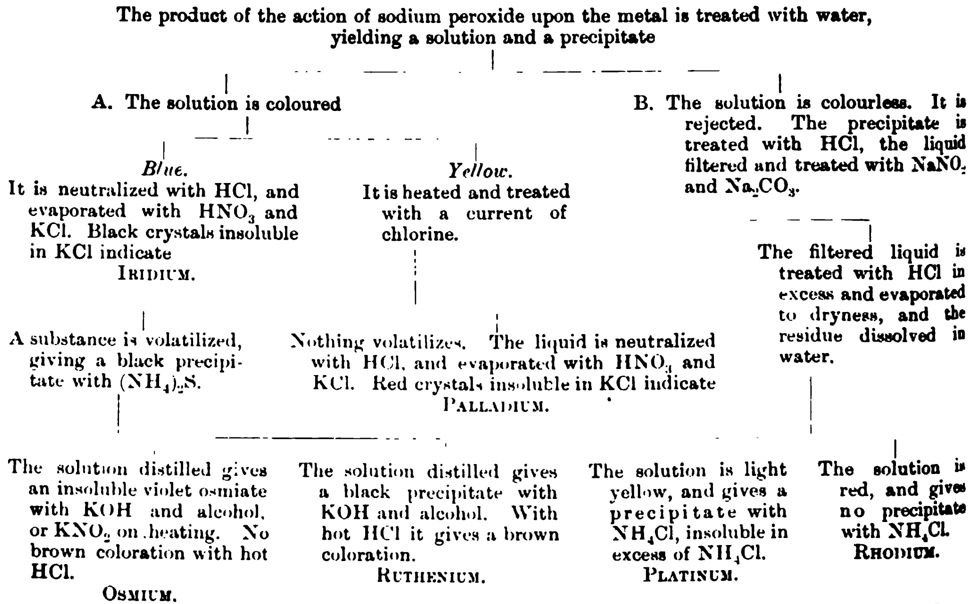
Palladium yields an alkaline palladate soluble in water. On neutralizing the yellow solution with hydrochloric acid and then evaporating it after the addition of potassium chloride and nitric acid, ruby red crystals of potassium chloro-palladate, insoluble in a saturated solution of potassium chloride, are formed.

Iridium is converted into a basic iridate, $IrO_3 4Na_2O$, which on treatment with water yields a blue solution. On adding a slight excess of hydrochloric acid to this solution, and evaporating after the further addition of potassium chloride and a little nitric acid, black crystals of potassium chloro-iridate are obtained. These are also insoluble in a saturated solution of potassium chloride.

Platinum is converted into the insoluble sodium platinate, which remains on the filter with the nickel oxide. The precipitate is dissolved in strong hydrochloric acid, the greater part of the acid subsequently evaporated, sodium nitrite added until the liquid is neutral, then sodium carbonate, and the liquid boiled and filtered. Nickel carbonate remains on the filter, whilst the filtrate contains the double nitrite of platinum and sodium. On evaporating this filtrate to dryness after the addition of hydrochloric acid, the residue dissolved in water and treated with ammonium chloride gives the characteristic crystals of ammonium chloro-platinate.

Rhodium is converted partially into the dioxide, RhO_2, soluble in hot concentrated hydrochloric acid, and partially into a sesquioxide, remaining undissolved. The filtrate obtained by treating the mass with hot hydrochloric acid will also contain the nickel. It is treated in the same manner as the platinum solution, and filtered from the nickel carbonate. On heating the filtrate with an excess of hydrochloric acid it becomes rose-coloured from the formation of the double chloride, $Rh_2Cl_6.6NaCl$, but does not yield a precipitate with ammonium chloride.

The following scheme shows the course to be followed in a systematic qualitative analysis:



The product of the action of sodium peroxide upon the metal is treated with water, yielding a solution and a precipitate

A. The solution is coloured

Blue.
It is neutralized with HCl, and evaporated with HNO_3 and KCl. Black crystals insoluble in KCl indicate
IRIDIUM.

Yellow.
It is heated and treated with a current of chlorine.

A substance is volatilized, giving a black precipitate with $(NH_4)_2S$.

Nothing volatilizes. The liquid is neutralized with HCl, and evaporated with HNO_3 and KCl. Red crystals insoluble in KCl indicate
PALLADIUM.

The solution distilled gives an insoluble violet osmiate with KOH and alcohol, or KNO_2 on heating. No brown coloration with hot HCl.
OSMIUM.

The solution distilled gives a black precipitate with KOH and alcohol. With hot HCl it gives a brown coloration.
RUTHENIUM.

B. The solution is colourless. It is rejected. The precipitate is treated with HCl, the liquid filtered and treated with $NaNO_2$ and Na_2CO_3.

The filtered liquid is treated with HCl in excess and evaporated to dryness, and the residue dissolved in water.

The solution is light yellow, and gives a precipitate with NH_4Cl, insoluble in excess of NH_4Cl.
PLATINUM.

The solution is red, and gives no precipitate with NH_4Cl.
RHODIUM.

C. A. M.

A Note on the Determination of Molybdenum in Steel. George Auchy. (*Journ. Amer. Chem. Soc.*, xxiv., 273.)—Not more than 1·308 grammes of the steel are treated with a large excess of strong nitric acid, with the addition of a little potassium chlorate if chromium is present; the nitric acid is evaporated off, the residue boiled with strong hydrochloric acid, evaporated to dryness to separate silica, taken up with strong hydrochloric acid, and the solution evaporated to the first appearance of a scum. To the solution 5 c.c. hydrochloric acid, diluted to 20 c.c. with water, are added, and after solution is complete the volume of the liquid is made up to 50 c.c. with water, and the solution poured little by little, with constant shaking, into a solution of 20 grammes caustic soda in 100 c.c. water, contained in a 300-c.c. flask. The liquid is made up to the mark with water, mixed, and 250 c.c. are filtered off, acidified with sulphuric acid, boiled down to less than 100 c.c., reduced with zinc, and titrated with permanganate. A blank must be made on a steel free from molybdenum, the same amount of hydrochloric acid and of chromium being present as in the actual determination. Most of the chromium is precipitated with the iron, but a small amount remains in solution. The method gives good results, the caustic soda separation being sufficiently exact for all practical purposes.

A. G. L.

The Analysis of Metallic Molybdenum and its Lower Oxides. C. Friedheim and M. K. Hoffmann. (*Berichte*, 1902, xxxv., 791-795.)—The pure molybdenum dioxide used by the authors in their experiments was prepared by reducing molybdenum trioxide for five to seven hours in a current of hydrogen at a temperature of 450° C., and then heating the mixture of dioxide and unreduced trioxide to a dull red heat in a current of dry hydrochloric acid gas so as to volatilize the trioxide in the form of chloride.

For the determination of the molybdenum dioxide in an insoluble molybdenum oxide the substance is slowly heated to the boiling-point with a sufficient quantity of a 10 per cent. solution of ferric ammonium sulphate and twice that amount of dilute sulphuric acid (1 : 5).

The molybdenum is dissolved in accordance with the equation

$$Fe_2(SO_4)_3 + MoO_2 + H_2O = 2FeSO_4 + H_2SO_4 + MoO_3,$$

and by titrating the amount of ferrous salt with standard permanganate the quantity of molybdenum dioxide can be obtained.

In the case of an oxide containing lower oxides of molybdenum soluble in water the substance is treated with an excess of acid potassium permanganate solution, and the excess titrated with standard oxalic acid solution.

Metallic molybdenum may be analysed by the ferric ammonium sulphate method described above, or the following indirect gravimetric method may be used: The metal, or the compound containing lower oxides, is heated for about an hour, with continual shaking on the water-bath, with a very dilute ammoniacal solution of silver sulphate. After standing for twelve hours, air being excluded, the separated silver is collected on a filter, washed with a very slightly ammoniacal 10 per cent. solution of ammonium nitrate, dissolved off the filter by means of dilute nitric acid, and determined either gravimetrically or volumetrically.

C. A. M.

Colorimetric Estimation of Sulphur in Cast Iron. T. Naske. (*Stahl und Eisen*, 1902, xxii., 333; through *Chem. Zeit. Rep.*, 1902, 92.)—Lindlay has described a process for the estimation of sulphur which depends on the fact that in presence of ferric chloride and sulphuretted hydrogen *p*-phenylenedimethyldiamine hydrochloride is converted into methylene blue. The sulphur of the iron is therefore to be liberated with hydrochloric acid, the gas passed into sodium hydroxide, and a colorimetric operation carried out. Naske, however, objects to this method that acid produces methyl sulphide as well as sulphuretted hydrogen, that on absorption in caustic soda normal sulphide, hydrosulphide, and polysulphides are formed, that the hydrogen sulphide is not quantitatively taken up, and that the sodium sulphide solution gradually changes into thiosulphate and sulphate. On the other hand, solutions of methylene blue are decomposed—*i.e.*, decolorized—by sulphuretted hydrogen and thiosulphate. Lindlay's process is accordingly only suitable for qualitative purposes, and the same remarks apply to the employment of Lauth's violet, which he has also recommended. F. H. L.

An Accurate Estimation of Sulphur in Iron by the Evolution Method. Harry E. Walters and Robert Miller. (*Proc. Eng. Soc. West. Pennsylvania*, xviii., 83.)—In this method 5 grammes of the sample are weighed out into a porcelain or copper boat, which is then placed in a porcelain or nickel combustion-tube, and heated to bright redness for fifteen to thirty minutes in a current of natural gas or hydrogen. After allowing it to cool in the current of gas, the sample is removed, transferred to a flask, and the hydrogen sulphide evolved as usual by means of hydrochloric acid (1 acid : 1 water). The evolved gases are absorbed in ammoniacal cadmium chloride solution and titrated with iodine. The results given by this method were checked against those given by the ordinary evolution method and by a gravimetric method, and are almost identical with the latter, the largest difference being 0·004 per cent. in about thirty determinations, whilst the ordinary evolution method in many cases gave very low results. For an ordinary pig-iron fifteen minutes' heating is sufficient; for samples containing titanium it is necessary to heat for half an hour. A. G. L.

The Determination of Sulphur in Iron Pyrites. R. Auzenat. (*Ann. de Chim. anal.*, 1902, vii., 93, 94.)—The following method is recommended as giving accurate results in a much shorter time, and with less attention to the evaporation, than those obtained by Lunge's method: About 0·5 gramme of the finely powdered mineral, previously dried at 100° C., is weighed into a flask, into which are then introduced successively 20 c.c. of a 10 per cent. solution of sodium chloride, 10 c.c. of hydrochloric acid, sp. gr. 1·18, and 10 c.c. of nitric acid, sp. gr. 1·5. As soon as effervescence has ceased, the flask is placed in an oil bath provided with a thermoregulator, and the liquid evaporated to dryness. Under these conditions the temperature can be raised to 120° C. without the liberation of free sulphuric acid.

The author's comparative determinations have shown that it is possible to save several hours in this way without fear of loss. C. A. M.

Rapid Method for Estimating Thorium in Monazite Sand. **F. Benz.** (*Zeits. für angew. Chem.*, 1902, xiii., 309.)—Half a gramme of the finely divided sand is intimately mixed in a platinum crucible with the same quantity of sodium fluoride and 10 grammes of potassium pyrosulphate and gradually heated. When the evolution of gas ceases the crucible is raised to a low red heat and kept at it for fifteen minutes. The melt is extracted first with dilute, then with concentrated hydrochloric acid. The solution is filtered. The filtrate is nearly neutralized with ammonia, taking care not to produce a permanent precipitate. It is raised to boiling, and 3 to 5 grammes of solid ammonium oxalate are added with stirring. The oxalates fall as a granular precipitate. A little ammonium oxalate solution is added to make sure that the precipitation is complete. After standing all night the oxalates are filtered off, then washed with as little water as possible into a porcelain basin. The filter is washed with hot concentrated nitric acid and water, and the liquid is evaporated almost to dryness. A few c.c. of concentrated nitric acid are added and then 20 c.c. of fuming nitric acid, the basin is covered with a watch-glass and placed upon the water-bath. When the effervescence ceases the liquid is evaporated to dryness; some water is added and again evaporated off. The residue is then dissolved in water and ammonium nitrate solution is added; the liquid is warmed to 60° or 80°, and 20 c.c. of distilled hydrogen peroxide (2 or 3 per cent.) are added. A precipitate of thorium peroxide is formed, which may be coloured yellow by minute traces of cerium peroxide. This is washed with hot water containing some ammonium nitrate, and when half dry is ignited in a platinum crucible and weighed as ThO_2.

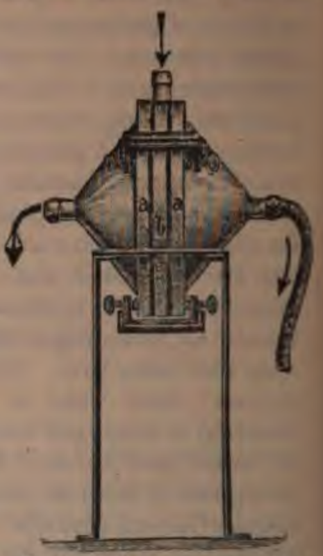
A. M.

A Rotary Cement Kiln for Use in the Laboratory. **E. D. Campbell.** (*Journ. Amer. Chem. Soc.*, xxiv., 249.)—The author describes a small rotary furnace for cement burning which gave very satisfactory results. The furnace itself consists of an 8-inch steam-pipe 32 inches long, the lining being made of four sections of magnesite tubes packed into the steel jacket with asbestos, the asbestos being mixed at one end for a distance of 2 inches with fire-clay and sodium silicate. The rotary is turned by means of a one-seventh horse-power electric motor, and heated by means of a Hoskins hydrocarbon burner. The temperature is controlled by means of a Le Chatelier thermocouple, the wires of which pass through a double-bored porcelain tube covered for a distance of 8 inches with platinum foil. A fixed couple is kept at a point 3·5 inches from the hot end, and the temperature at other points is taken by means of a moving couple. The hottest zone is at a distance of 6 inches from the discharge end. The temperature at this point can be raised up to 1,630° C., which is too high for most mixtures. About 4,500 grammes of material are usually used for one run, yielding 2,500 to 2,800 grammes clinker, at the rate of about 600 to 700 grammes clinker per hour from the first appearance of clinker to the time feeding is stopped.

A. G. L.

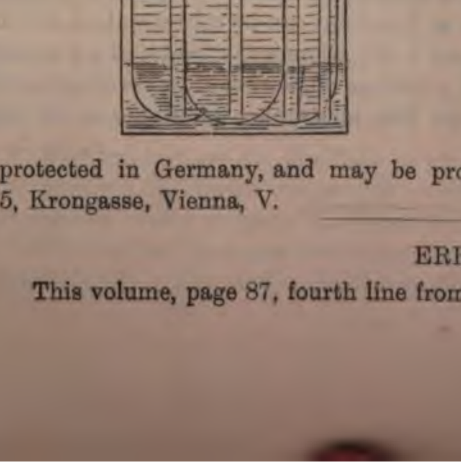
APPARATUS.

A Laboratory Filter-press. (*Chem. Zeit.*, 1901, xxv., 1162.)—This apparatus consists of a pair of porcelain cones, with leading tubes at the apices and with flat, perforated plates at the bases. Between them is clamped a porcelain ring or distance-piece, square in section, and provided with an entrance tube. To put the apparatus together, one cone is held point downwards on a suitable stand, a piece of filter-cloth or asbestos is laid on the perforated plate, then a rubber-washer is put in position, next the distance-piece, next a second cloth and rubber ring, and finally the other cone is applied, and the whole clamped up. In use it is supported as shown, the turbid liquid entering through a tube about 1·5 metres high to give the requisite pressure. The liquid then filters right and left through the cloths. To wash the precipitate, the apparatus is supported in the other position, the entry tube being closed, and water being forced in at one apex only. By adjusting a Liebig condenser round the pipe through which the thick liquor is being admitted, filtration may be conducted in the warm. Clearly the apparatus can be employed with suction instead of with pressure. It is patented, and may be obtained from Kaehler and Martini of Berlin, W.



F. H. L.

A New Potash Apparatus. A. Landsiedl. (*Oesterr. Chem. Zeit.*, 1902, v., 30.)—The construction of this apparatus is fully explained by the accompanying diagram. It will be seen that the stream of gas has to pass through the potassium hydroxide contained in the three inner vessels, and then through that in the outer bottle. The device is filled through *b* by suction, and emptied through *a* by blowing. By sucking sharply at *b* the whole of the potash may be drawn into the outer vessel and so mixed together, being then driven back into its proper position by a cautious aspiration at *a*. It is claimed for the apparatus that it is easy to clean, very compact, and that it stands firmly. It is protected in Germany, and may be procured from the firm of V. E. Zahradnik, 5, Krongasse, Vienna, V.



F. H. L.

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THE ANALYST.

JULY, 1902.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

A SPECIAL general meeting of the Society was held in the Chemical Society's rooms, Burlington House, on Wednesday evening, May 21, when a discussion took place on the Report of the Joint Committee appointed by the Society of Chemical Industry and the Society of Public Analysts to consider the question of the determination of minute quantities of arsenic. Sir William Church and Dr. G. S. Buchanan, of the Royal Commission on Arsenical Poisoning, Mr. Graham Aldous, and a number of members of the Society of Chemical Industry, were present. The President (Dr. J. Augustus Voelcker, M.A., B.Sc.) occupied the chair.

The minutes of the previous meeting having been read and confirmed, a certificate of proposal for election to membership in favour of Mr. Alfred Howard, A.R.C.S., A.R.S.M., was read for the second time.

The monthly meeting of the Society was held on Wednesday evening, June 4, in the Chemical Society's rooms, Burlington House. The President (Dr. J. Augustus Voelcker, M.A., B.Sc.) occupied the chair.

The minutes of the previous meeting were read and confirmed.

A certificate of proposal for election to membership in favour of Mr. Peter Macdonald, Apartado No. 17, Saltillo, Mexico, assayer to the Mazapil Copper Co., Mexico, was read for the first time.

Messrs. M. Wynter Blyth, Alfred Howard, and Walter F. Sutherst were elected members of the Society.

The following papers were read: "Note on the Examination of Water from some Typhoid-polluted Wells," by S. Rideal, D.Sc.; "The Estimation of Platinum in Alloys," by Percy A. E. Richards; "The Electrolytic Detection and Estimation of Arsenic," by E. J. Bevan; and "The Action of boiling Hydrochloric Acid on Arsenic Acid," by Otto Hehner.

DISCUSSION ON THE REPORT OF THE JOINT COMMITTEE OF THE SOCIETY OF CHEMICAL INDUSTRY AND PUBLIC ANALYSTS ON THE DETERMINATION OF MINUTE QUANTITIES OF ARSENIC.

(*Held at the Meeting, May* 21, 1902.)

AFTER some preliminary remarks by the President, Mr. **HEHNER**, Chairman of the Joint Committee of the two Societies, stated that it had been found that the process of purification of hydrochloric acid prescribed by the Committee, although fully answering in the majority of cases, had not been infallible in freeing acid from arsenic.

It had been found that it was better not to dilute the acid as prescribed, but to use the strong acid of commerce, to add bromine and an excess of sulphurous acid to it, and to distil off and reject about one-fifth, the remainder of the distillate being then perfectly free from arsenic, even if crude yellow hydrochloric acid had been employed.

It had further been found that zinc could be freed from arsenic by treating it in its molten condition with sodium, and removing the sodium as completely as possible by subsequent air oxidation.

The discussion was opened by Dr. **THOMAS STEVENSON**, who thought the mode of purification of hydrochloric acid as recommended by the Committee somewhat too cumbrous, and expressed his opinion that, by distilling off a portion and taking the remainder, acid sufficiently free from arsenic could always be obtained. He further objected to the presence of the hydrobromic acid, as this might form free bromine, which might be a source of error. For the testing of commercial products he preferred the Reinsch method. He admitted that it was capable of detecting only one part of arsenic in three or four million parts, with a limit of about one-fiftieth of a grain per gallon in the case of beer. If arsenic were present in organic combination, neither the Reinsch nor the Marsh test would discover it, and in such cases the destruction of organic matter was a necessity.

Mr. **ALLEN** criticised the Committee's process of hydrochloric acid purification, and preferred oxidizing any arsenic which might be present by potassium permanganate or bichromate of potash, and distilling, rejecting the first small portion. The different methods of sulphuric acid purification answered, he thought, well. It was a matter of considerable trouble to obtain zinc answering all requirements, and he expressed his strong conviction that a small quantity of iron must be present with it in order to give an equal flow of hydrogen gas and a comparable brown mirror. At the same time, the presence of too much iron must be avoided, or the zinc becomes insensitive.

Mr. **A. R. LING**, referring to Dr. Stevenson's censure of the method of purifying hydrochloric acid, pointed out that pure hydrochloric acid was liable to contain free chlorine. As regards the treatment of zinc by means of sodium, it was very important to remove all the sodium, preferably by baking the zinc in a muffle.

Mr. **R. BODMER** had failed to obtain indications of arsenic both by the Marsh and Reinsch methods in the case of cacodylates. He had also failed to obtain good results with the Committee's basic process for destruction of the organic matter.

Mr. JULIAN BAKER referred to the difficulty of testing the dust from malt-kilns, and recommended that these should always be burnt with lime, and then tested by the Marsh process, and not directly.

Mr. WOOD-SMITH gave his opinion that the Committee's process was the only practicable one, and he considered that the use of the Reinsch process was fraught with danger to the brewer.

Dr. L. T. THORNE gave a description of his process for purifying hydrochloric acid,* already described by him to the Chemical Society. He had found that, starting with good zinc, one treatment with sodium was sufficient to remove all traces of arsenic. He had found no loss of sensitiveness, but gave the warning that no sodium should be left in the zinc. His experience was that with sugars he always obtained the same results with or without destruction of the organic matter. He considered the Marsh test to be infinitely preferable to the Reinsch.

Dr. P. SCHIDROWITZ thought the test to be, on the whole, a good one for all practical purposes. As regards the sensitiveness of either zinc or acid, he suggested that selenium might be one of the factors to be taken into consideration.

Dr. BERNARD DYER demurred somewhat to the small quantity of beer to be taken for the test. He preferred to employ a larger quantity. He used the Committee's process with hydrochloric acid for all sorting purposes, but in cases requiring further investigation he worked upon a quantity of 70 c.c. or more, destroying the organic matter by acid treatment.

Mr. BENJAMIN E. R. NEWLANDS pointed out that the basic method of destruction of organic matter had been devised by himself, and was the subject of a patent for the retention of arsenic in coke for malting purposes.

Mr. A. J. MURPHY preferred to ascertain approximately the amount of arsenic by the Committee's process, and then to run three parallel tests—one a blank, the second a blank with the addition of a quantity of arsenious acid corresponding with the result obtained in the first test, and the third with a known quantity of the substance to be tested, so that in every case he obtained a fresh standard for comparison.

Messrs. L. BRIANT and A. J. STAREY made suggestions bearing upon the heating of the tube and the cooling of that portion of it where the mirror was expected to be deposited.

Mr. HEHNER, in summing up, showed that, with one single exception, the members who had expressed their opinions had been unanimous in favour, at least, of the principle underlying the Joint Committee's method, and that the discussion had been mainly concerned with the consideration of small details, which in themselves did not seriously modify the method in question.

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* See abstract, this number, p. 233.

ALKALINE WATERS FROM THE LOWER GREENSAND.

By W. W. Fisher, M.A., F.I.C.

(*Read at the Meeting, April* 9, 1902.)

In a paper recently read before the Society entitled "On Alkaline Waters from the Chalk" it was pointed out that alkaline waters are obtained not only from the chalk where it is covered by thick beds of London clay and tertiaries, but also from other porous rocks similarly covered. (See Analyst, August, 1901.)

Although abnormal chalk waters have often been discussed, little attention has been paid to similar waters in lower greensand or Portland beds underlying the gault, which belong to a much earlier epoch in geological history.

In those localities where the lower greensand is exposed it is very porous, and absorbs a large proportion of the rainfall, and yields generally good waters, many of which, containing little lime, are soft in character. In Table I. a few typical examples of such waters are given, from which it will be seen that the total solids are sometimes very small, with low figures for chlorides and saline constituents, the proportion of organic matter also being quite small. Notable quantities of nitrates are often present, and occasionally the proportion of iron is large and causes inconvenience.

Beneath the lower greensand, and practically continuous with it as regards water-bearing characters, are the Portland beds, which in Berks and Oxfordshire are often sandy in character, with occasional masses or beds of limestone rock. Typical waters from these beds are given in Table II., and it will appear that they do not differ in any important respect from other limestone waters. Inasmuch as a thick bed of Kimmeridge clay underlies the Portland beds, no water can percolate downwards, and in most localities water is easily obtained by wells of moderate depth.

In the counties of Oxford and Buckingham both the greensand and Portland beds, having a slight dip to the south-east, pass gradually beneath the gault clay, and considerable areas of these water-bearing strata are thus buried. Over these districts the water is obtained by borings often 200 feet in depth. The gault continues through Berkshire into Wiltshire, but only as a narrow strip, and where the greensand has been removed by denudation, and the Portland beds are absent, the gault lies directly upon the Kimmeridge clay, and for fifteen miles the two formations are in contact. Near Farringdon, Berks, the greensand reappears, but further west becomes narrow and of little importance in respect of water supply.

Opportunities have offered from time to time of examining waters obtained by borings through the gault, and the results of the analysis of some interesting samples are embodied in Table III., a more detailed account of the saline constituents being set out in Table IV. in respect of five samples.

These samples are materially different in character from those obtained from the uncovered beds; they contain considerable quantities of saline constituents, chiefly alkaline chlorides, sulphates, and carbonates; the proportion of nitrogen as nitrates is small, but, as in many deep-well waters, distinct amounts of ammonia are present.

If we compare the waters in Table IV. with each other very striking differences

TABLE I.—WATERS FROM THE LOWER GREENSAND (UNCOVERED GREENSAND).

(*Results in Grains per Gallon.* 1 *Gallon* = 70,000 *Grains.*)

No.	Locality.	Depth in Feet.	Total Solids.	Chlorine in Chlorides.	Nitrogen in Nitrates.	Saline Ammonia.	Albu-menoid Ammonia	Oxygen absorbed, etc. 3 hours.	Total Hardness.	Remarks.
1132	Bagley, Berks	25	19·3	0·9	·640	·004	·003	·021	13·5	
1269	Boars Hill, ,,	38	6·0	1·0	·210	0	·004	·013	—	Soft.
2046	,, ,,	—	20·4	1·3	·396	·003	·008	·007		
2190	Faringdon, ,,	—	30·8	1·1	·85	·001	·005	·012	19·1	Permanent hardness, 5·7.
2582	Near Faringdon, ,,	—	37·0	4·1	·07	·003	·008	·018	—	Hard.
2583	Uffington, ,,	—	28·0	2·9	·014	·030	·012	·182	—	Soft, contains iron.
2563	Wantage, ,,	—	32·0	1·3	·385	·002	·006	·013	—	Hard.
1341	Shotover, Oxon	—	10·6	1·4	·490	0	·001	0	—	Soft.
2052	,, ,,	Spring	19·9	1·6	·315	·003	·007	·042	—	
1307	Nuneham, ,,	—	17·3	1·5	·175	·001	·006	·023	—	
2509	,, ,,	90	23·5	2·9	·210	·006	·004	·019	—	Soft.
1859	Clifton Hampden, ,,	—	24·0	1·4	·497	·001	·005	·014	—	
2485	Great Brickhill, Bucks	Spring	14·0	1·2	·497	·003	·001	·014	—	Soft.
1941	Godalming, Surrey	,,	20·4	1·2	·028	·003	·001	·005	16·0	Contains a little iron.

TABLE II.—WATERS FROM THE PORTLAND BEDS (UNCOVERED).

(*Analysis in Grains per Gallon.*)

No.	Locality.	Depth in Feet.	Total Solids.	Chlorine in Chlorides.	Nitrogen in Nitrates.	Saline Ammonia.	Albu-menoid Ammonia.	Oxygen absorbed, etc. 3 hours.	Total Hardness.	Remarks.
2519	Radley, Berks ...	80	29·6	1·9	·014	·001	·003	·011	—	Hard.
1518	Iffley, Oxon ...	—	32·0	1·0	·546	0	·003	·022	—	,,
2363	Cuddesdon, ,, ...	—	56·0	1·2	·028	·003	·009	·070	—	Hard, excess $CaSO_4$ from Kimmeridge clay.
1127	Stowe, Bucks ...	Spring	23·2	1·4	·154	0	·012	·043	18·0	
1969	Brierton, ,, ...	12	38·0	1·8	·532	0	·006	·030	—	Hard.
2082	Quainton, ,, ...	Spring	27·2	1·0	·448	·001	·003	0	22·6	,,

TABLE III.—WATERS FROM LOWER GREENSAND (OR PORTLAND BEDS) BELOW GAULT CLAY.

(*Analysis in Grains per Gallon.*)

No.	Locality.	Depth in Feet.	Total Solids.	Chlorine in Chlorides.	Nitrogen in Nitrates.	Saline Ammonia.	Albumenoid Ammonia.	Oxygen absorbed, etc. 3 hours.	Total Hardness.	Remarks.
3008	Warborough, Oxon	177	546·0	190·7	·05	·252	·006	·044	—	Water-level 6 ft. from surface.
2749	Newington, „	85(?)	414·0	148·5	·014	·072	·003	·018	—	„ „ 40 ft. „ „
2684	Stadhampton, „	52	236·0	48·3	·021	·072	·006	·124	32·9	„ „ 47 ft. „ „
3849	Near Thame, „	96	77·2	12·1	·028	·037	·003	·032	3·3	„ „ 36 ft. „ „
3743	Wheatfield, „	211	79·0	9·7	·014	·028	·005	·049	3·5	„ „ 35 ft. „ „
3801	Cheddington, Bucks	213	26·6	1·9	·014	·032	·003	·024	9·7	„ „ 113 ft. „ „
3230	Rowsham, „	12	95·0	2·5	·021	·005	·005	·008	Hard	„ „ 3 ft. „ „
3283	Near Cambridge ...	134	215·0	19·9	·014	·071	·024	·160	„	Excess $CaSO_4$

TABLE IV. (*Grains per Gallon.*)

No. ... Locality	3008 Warborough.	2749 Newington.	2684 Stadhampton.	3849 Near Thame.	3743 Wheatfield.	3801 Cheddington.
Chlorine	190·7	148·5	48·3	12·1	9·7	1·9
Sulphuric acid, SO_3	126·87	88·2	—	4·2	4·66	1·91
Carbon dioxide, CO_2...	4·80	7·3	6·7	20·5	21·56	8·08
Soda, Na_2O	241·19	188·0	—	40·3	[48·65]	[4·16]
Magnesia, MgO	7·14	5·88	—	·5	·50	1·54
Lime, CaO	17·50	9·8	—	·84	1·40	6·30
Silica, SiO_2	1·09	—	—	·42	2·1	2·21
					Fe_2O_3·84	Fe_2O_3·21
Sodium chloride	314·25	244·7	79·3	19·94	15·92	3·13
Sodium sulphate	171·14	133·8	—	7·45	8·05	3·38
Sodium carbonate	—	—	—	45·30	48·22	2·48
Magnesium sulphate	8·20	17·48	—	—	—	—
Magnesium carbonate	9·19	—	—	1·05	1·07	3·22
Calcium carbonate	—	15·98	—	1·50	2·50	11·25
Calcium sulphate	42·50	2·07	—	—	—	—
Total solids	546·37	414·04	236 0	77·28	79·0	26·6

may be noted. In the first pair from Warborough and Newington large quantities of sodium chloride and sulphate are present, with smaller quantities of magnesium and calcium salts. The solid residues were alkaline in reaction, but no free sodium carbonate was found. In the next pair of wells, about eight miles distant from the former, we find at Wheatfield and near Thame, although some sodium chloride and sulphate are present, sodium carbonate is the most abundant constituent. A third well near Thame yields water almost identical in composition, and I may mention that both the Thame wells have been under my observation for twenty years, and the waters have hardly changed in character during that period.

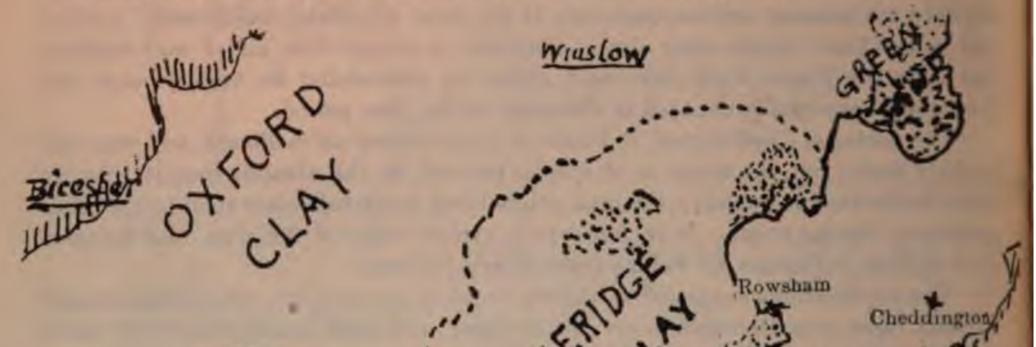
The boring at Cheddington, in Bucks, is about sixteen miles distant, and was only recently made, but the water is of special interest, as the alkaline constituents are much diminished in quantity, the total solids being remarkably less than in the other samples of similar origin. It is, however, a typical water of the class, and contains some sodium carbonate, as well as chloride and sulphate.

The explanations suggested at various times to account for soft alkaline waters in chalk have been discussed in my former paper, and need not be referred to again, except to point out that the view that the alkaline waters are derived from the sea by infiltration is clearly out of the question in respect of these samples. The hypothesis which was dismissed as untenable in respect of the chalk waters is still more improbable as an explanation of the peculiarities of lower greensand waters.

It seems beyond doubt that the mineral constituents of these waters are derived from the beds from which they are drawn—that is, from the greensand and Portland beds—and not from the clay or any other external source.

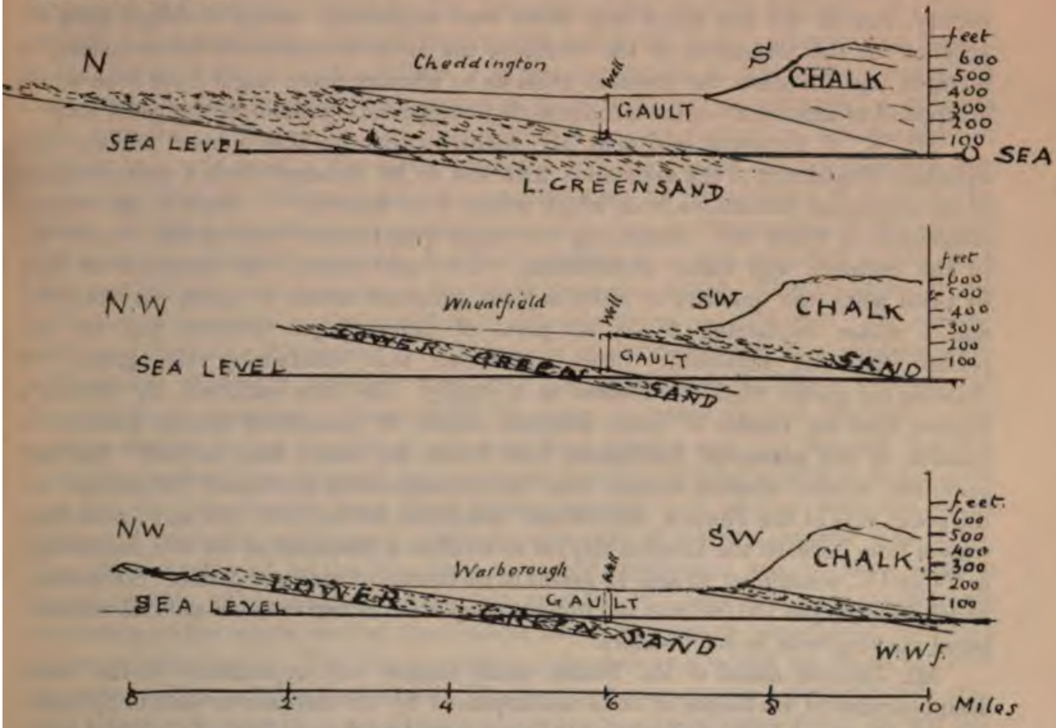
The exposed parts of these beds, as of other porous and permeable formations, have for long ages beyond reckoning been exposed to continual percolation, and by natural discharges from springs the more soluble constituents, especially the alkaline salts, have been eliminated. On the other hand, where such beds are covered by extensive clay deposits percolation must be slow and difficult, and, unless natural outlets exist, no removal of soluble salts can take place. It must further be remembered that most of these waters from borings in the gault have travelled long distances beneath the clay from their gathering grounds—possibly three or four miles—which means 15,000 or 20,000 feet of material to be traversed at an extremely slow rate, the conditions thus in the highest degree favouring the extraction of soluble matters.

Yet, accepting these considerations, it is not at first apparent why there should be such large differences between the Warborough, Wheatfield, and Cheddington samples, but a study of the surface contours suggests that they may arise from differences in level. We find that at Warborough, in the Thames Valley (close to the river), the surface level is 160 feet above Ordnance Datum, but at Wheatfield 257 feet (nearly 100 feet higher); while towards Cheddington the surface gradually rises to 342 feet at the site of the well, and even above 400 feet in the neighbourhood. Deducting the depth of boring (177 feet at Warborough), we find the bottom of the gault is 17 feet below sea-level at this locality, whereas at Wheatfield (the depth being 211 feet) the bottom of the clay and the top of the greensand is 46 feet above Ordnance Datum. The depth near Thame is probably much the same, while at Cheddington the elevation similarly is 129 feet at the base of the clay. The greensand is full of water, which rises at all wells, and, taking the water-levels in Table III., we find the water surfaces respectively are 154 feet O.D., 222 feet O.D., and 229 feet above O.D. at Cheddington. Clearly this water may slowly travel along the slightly inclined strata, and escape by any natural outlet at a lower level where springs exist. The northwestern margin of the gault is in many places below this level, and an escape for the waters accordingly exists. At Warborough it is not possible for the buried waters to find a natural escape, but probably near Thame some discharge takes place, which even in limited quantity permits some movement onwards of the underground waters, and accounts for the intermediate character of the samples. It is



worthy of note that the Thames Valley is actually the lowest part of the gault which rises gradually through Berkshire until it reaches a considerable elevation.*

I was fortunate in obtaining a specimen of a core from the lower greensand at Wheatfield for examination. It contained an appreciable quantity of sodium carbonate, with traces only of alkaline sulphate and chloride. When finely ground and thoroughly extracted with water, the sodium carbonate estimated by titration was 0·25 per cent. No evidence of the presence of silicate of soda was obtained in the aqueous solution.



Geological maps, superficial and sectional, of the district whence these waters were obtained are appended.

My best thanks are due to Mr. A. W. Ellis for his co-operation, and for most of the analyses of the mineral constituents of the deep-well waters.

The President having invited discussion,

Mr. F. J. Lloyd observed that the water from Uffington, No. 2583, seemed to be quite different from all the other waters referred to in Table I., and he would certainly think from the figures that that water was not free from contamination. The next question that arose in his mind was as to the source of these comparatively large quantities of saline ammonia. It would appear, if the nitrates had been reduced to ammonia, that larger quantities of saline ammonia, with correspondingly smaller quantities of nitrates, would be found as the depth of the bed increased; and the figures given supported that view to a certain extent, but not altogether, because in the water from Cheddington (Table III.) the nitric nitrogen was only slightly less than that in the next sample, from Rowsham, although the water-level in the latter case was only 3 feet from the surface, as against 113 feet in the former case. Of course, it may have been that the beds were entirely different, although in the same county, but he did not think that there was sufficiently strong evidence here to account for the formation of the whole of the saline ammonia by the reduction of nitrates, and therefore the question arose as to whether there might have been some compound of ammonia—some ammonia-clay compound—present in the gault clay.

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* A parallel case is found in Essex, where the waters from deep wells in chalk beneath the London clay are alkaline only where the chalk is much below Ordnance Datum, but calcareous when derived from the more elevated chalk under the clay in the northern parts of the county, and where natural drainage is accordingly possible.

Mr. W. T. Burgess said that Mr. Fisher's work on this subject afforded an excellent illustration of the assistance that was to be obtained from a consideration of the geological formations from which waters were derived. Some of the waters mentioned in Table III., containing very large quantities of total solids, would be in the ordinary way quite undrinkable. He could detect the presence of salt by taste when the quantity of chlorine just exceeded about 15 parts per 100,000, and a water containing 30 or 40 parts of chlorine per 100,000 was to his palate distinctly brackish. Such waters as that containing 190 grains of chlorine per gallon would be useless as a supply. He fully endorsed Mr. Fisher's opinion that the results of water analyses should be considered almost entirely in relation to the geological formations from which the waters were derived. He had examined several alkaline waters from the corresponding geological formations on the south side of the Thames, but did not remember having met with any (excluding waters from beneath the London clay) of so alkaline a character as the two mentioned in Table IV., containing 45 and 48 grains of carbonate of soda per gallon. Something under 30 parts per 100,000 was the highest proportion of carbonate of soda he remembered meeting with in such waters.

Mr. Jenkins asked if Mr. Fisher could suggest any explanation of the total disappearance of carbonate of soda accompanied by an increase in sodium chloride and in sodium sulphate in the case of the waters referred to in Table IV., as the beds went down towards the south-west.

The President (Dr. Voelcker) said that, in dealing with waters from the neighbourhood of Woburn, he had met with great variations in the quality of the water drawn from different springs. These variations, however, would be to a large extent explained by the facts which Mr. Fisher had put forward. The farm with which he was concerned was on the lower greensand formation, and it would be imagined that the waters obtained from the greensand itself would not be of a particularly hard nature, and would not contain large quantities of chlorides or sulphates; but, as a matter of fact, although in some parts of Buckinghamshire, just on the borders of Bedfordshire, such waters were met with as that at Great Brickhill (Table I.), which contained only 14 grains of total solids per gallon, nevertheless, the water obtained at the farm to which he was referring, only about three miles from Great Brickhill, contained large quantities of chlorides and sulphates. This constituted a considerable difficulty in the way of obtaining a suitable supply of water for the cottages and farms on the Bedford estate. He agreed with Mr. Lloyd in thinking that the Uffington water required some explanation; and the large quantity of saline ammonia in the sample from Warborough would lead one to wish for further knowledge in regard to that supply also. Possibly the explanation might lie in the fact that the water-level was only 6 feet from the surface.

Mr. **Fisher**, in reply, said that it seemed to him quite open to argument, on the figures of the analysis, whether the Uffington water was an entirely unpolluted sample. The water was obtained from a spot which the local authority had chosen in looking for a supply for the village. As was mentioned in the table, it contained iron, being, in fact, so highly ferruginous as to be unfit for the purpose for which it was intended. Although it was drawn from a porous formation, the quantity of nitrates in it was small. The saline ammonia may have been, and possibly was, due to the fact that the well had been only recently made for the purpose of proving the water. The quantity of oxygen absorbed was high, but, as the quantity of albuminoid ammonia was relatively moderate, the high quantity of oxygen absorbed might have been partly due to the presence of ferrous iron in the water. The question of the formation of ammonia by the reduction of nitrates was a complex one. The investigations of Professor Warington on the subject of denitrification by organisms were well known. Probably, however, there was not much bacterial life in existence at the depths in question. There might be some ammonia derived originally from organic remains in the clay which had never been washed out. It was not possible to explain entirely the origin of the ammonia, but the point he wished to insist upon was that, in these waters from great depths beneath beds of clay, ammonia was a constantly occurring constituent, while the quantity of nitrates present was small. The point mentioned by the President in regard to Woburn was, perhaps, to some extent capable of explanation by the fact that the drift-clay covered a considerable proportion of the estate, so that there could be no downward percolation, but only horizontal circulation. The Warborough sample obviously would not be fit to drink on account of its saline constituents. The boring, however, was a perfectly new one, and, although he could not say exactly where the ammonia came from, he did not think it likely that the water had become seriously contaminated.

THE DETECTION AND ESTIMATION OF MINUTE QUANTITIES OF SULPHURETTED HYDROGEN IN COAL-GAS.

By W. J. Dibdin, F.I.C., and R. G. Grimwood, F.I.C.

(Read at the Meeting, April 9, 1902.)

Owing to a recent proposal to modify the test for sulphuretted hydrogen as used on the London gas supply, it became necessary to ascertain, as no published data could be found on the point, what was the best means of estimating small quantities of sulphuretted hydrogen in coal-gas, and what degree of delicacy could be attained in its detection.

The apparently sensitive colour reaction between sodium nitro-prusside and an alkaline sulphide was examined, but it was found that the sensitiveness was more apparent than real, a much larger quantity than is ever likely to be present in purified coal-gas being required to produce any colour.

Recourse was therefore had to the coloration of lead salts, which is the system at present in use. Although it is well recognised that this reaction is a very delicate one, it is somewhat surprising to find how extraordinary its delicacy is; in fact, instead of there being any difficulty in detecting minute traces, the difficulty is to modify the test so that infinitesimal traces are not detected.

An attempt to produce a titrateable solution of a lead salt by passing through it gas containing sulphuretted hydrogen was unsuccessful, owing to the colour which was also produced by tarry matters in the gas. The coloration of paper soaked in a solution of a lead salt was next examined, papers soaked in various strengths of lead salts in solution being compared. From the results of this comparison it was decided to adopt a paper prepared by moistening with an approximately 12 per cent. solution of lead acetate, the paper whilst still wet being placed for a few minutes in an atmosphere of ammonia gas. Papers prepared in this way are used in London for testing the purity of the gas supply.

For several reasons it was found preferable to use these papers dry, considerable difficulty being found in distinguishing when the colour first appeared on a wet paper, and also because when wet they were unworkably sensitive.

The apparatus employed for preparing the sulphuretted hydrogen-contaminated gases consisted of a glass reservoir of known capacity, into which measured quantities of sulphuretted hydrogen could be introduced. As soon as the sulphuretted hydrogen had thoroughly diffused the contaminated gas was examined by expanding a toy balloon inside the reservoir, and thus forcing the gas through a small bore tube, so arranged that the gas as it issued from it impinged on a lead paper prepared as already described. The following illustration shows the apparatus employed.

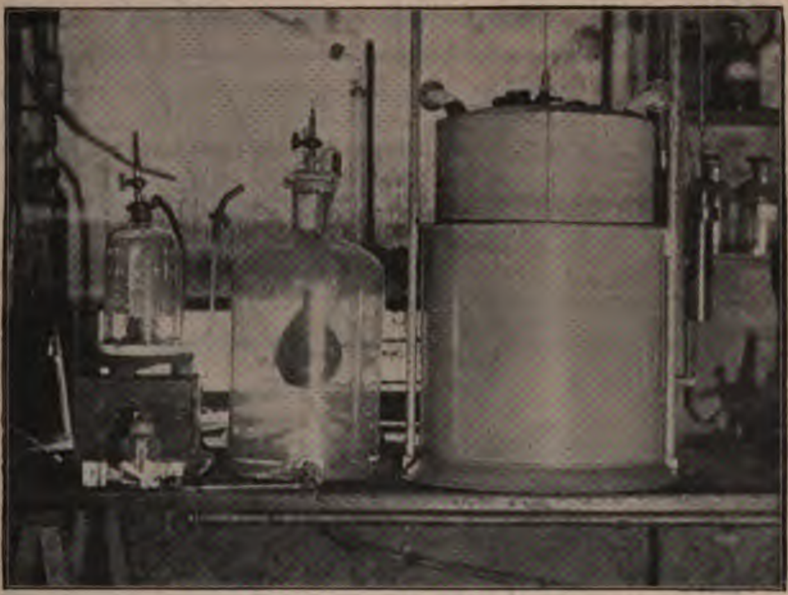
The reservoir, the capacity of which is 15,500 c.c. when the balloon is deflated, is fitted at the neck with an I.R. stopper pierced with three holes, one fitted with a cock of sufficient bore to pass at least ten feet of coal-gas per hour under the ordinary gas-supply pressure; the second with a glass tube, reaching about one-third down the reservoir, to the end of which is fitted gas-tight an I.R. balloon capable of being expanded so as to fill the reservoir; and the third with a narrow bore glass tube leading to the apparatus where the examination is made.

This consisted of a bell-jar standing in a mercury seal, and fitted at the top with a glass tube, which provides a secondary gas supply, and a cock and a burner, which gives a jet flame. By means of a cock or screw-clip on the inlet tube the height of the flame can be adjusted in this burner so that the gas burns at half a foot per hour.

At the bottom of the reservoir two tubes are fitted through an I.R. stopper, one of which connects with the gas-supply, and the other, which is of small bore, is fitted with a cock or clip.

To ascertain what amount of sulphuretted hydrogen could be detected in a coal-gas, a known volume of sulphuretted hydrogen was introduced through the capillary tube at the bottom of the reservoir, and allowed to diffuse through the gas. This, as a rule, took from thirty to forty-five minutes. When diffusion was complete, the balloon was partially inflated, thus forcing some of the gas into the apparatus containing the lead paper, which was fixed in such a way that the gas impinged directly on it. In order to insure the thorough diffusion of the sulphuretted hydrogen a second test was always made with the apparatus connected to the bottom of the reservoir and the spots on the two slips of paper compared.

If sulphuretted hydrogen was in this way detected, a measured volume of the contaminated gas was withdrawn from the reservoir and the remainder washed out by a current of coal-gas, the gas being burnt from the cock at the top. As soon as the gas in the reservoir was free from sulphuretted hydrogen, the measured volume of contaminated gas was replaced, diffusion allowed to take place, and the contents again examined. By this means it was possible to detect, within a reasonable time, one volume of sulphuretted hydrogen in 1,000,000,000 of coal-gas, equivalent to approximately 1 grain of sulphuretted hydrogen in 600,000 cubic feet of coal-gas.



Having thus found the limit to which it could be detected by this means, an attempt was made to produce a standard set of colours corresponding to varying proportions of sulphuretted hydrogen.

Owing, however, to the extremely evanescent nature of the colour-spot, and to the difficulty of being sure the colours matched, this was abandoned in favour of what was found to be a more certain method.

By regulating the lead paper to a known distance from the nozzle, where the gas issues, and passing the gas at a half-foot per hour, it was found that the time required for the production of a colour was a constant for that proportion at that distance.

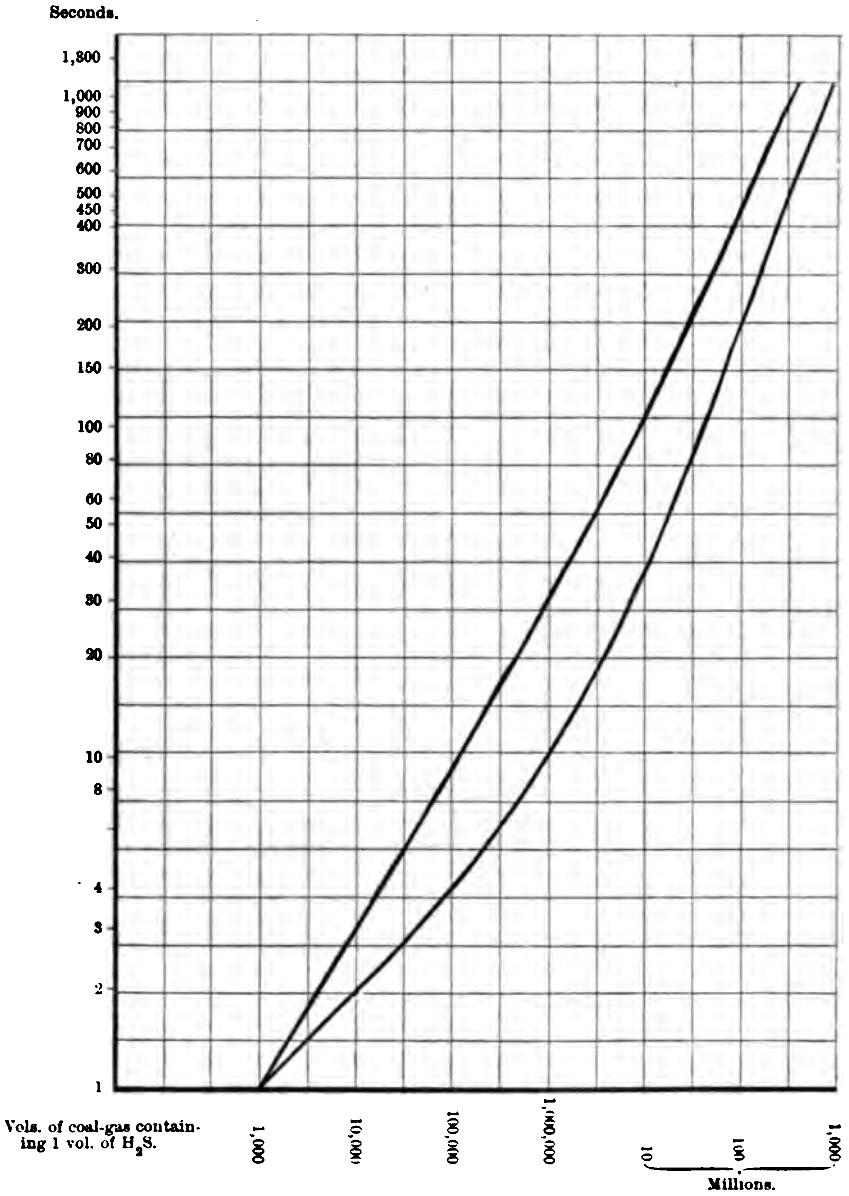


DIAGRAM SHOWING TIME REQUIRED TO DETECT VARYING QUANTITIES OF H_2S IN COAL-GAS.

The following table gives some of the mean results obtained by this method at different distances. The variation by different tests in any result was but slight.

Time taken to produce a Colour Spot on Lead Paper by Gas containing One Volume of Sulphuretted Hydrogen in

Distance of Lead Paper from Nozzle at which Gas issues.	1,000 volumes of Coal-Gas.	10,000 volumes of Coal-Gas. min. secs.	250,000 volumes of Coal-Gas. min. secs.	One million volumes of Coal-Gas. min. secs.	Ten million volumes of Coal-Gas. min. secs.	100 million volumes of Coal-Gas. min. secs.	1,000 million volumes of Coal-Gas. min. secs.	10,000 million volumes of Coal-Gas. mins.
Close to...	at once	0 2	0 5	0 8	1 7	5 0	11 30	more than 30 mins.
⅛ inch ...	,,	0 4	0 10	0 15	1 30	—	—	,,
¼ inch ...	,,	0 8	0 15	0 30	1 50	12 0	21 30	,,
½ inch ...	,,	0 12	0 18	—	2 20	—	—	,,

Two curves have been plotted on the results obtained, the ordinates representing the number of volumes of coal-gas containing one volume of sulphuretted hydrogen and the abscissæ time in seconds. The ordinates are plotted in geometrical progression, it being obviously impossible to plot them as an arithmetical progression, and consequently time is plotted in the same manner. Two curves have been plotted in order to show the importance of the distance factor.

The right-hand curve represents the results found when the lead paper was close to the point of issue of the gas, and the left-hand curve the results obtained when the paper was adjusted to a distance of ¼ inch from the nozzle.

DISCUSSION.

Mr. R. **ORCHARD** said that Dr. Rideal had experimented in a similar way to that described by Mr. Grimwood. In his experiment, using the gas referees' apparatus, Dr. Rideal had found that the delicacy of the official test was such that 1 part of sulphuretted hydrogen in five millions could be detected, passing 5 cubic feet of gas per hour. At first a gold-beater's skin balloon had been used. Subsequently a rubber balloon was tried, but it was found very difficult to free the rubber from sulphuretted hydrogen, and on allowing the apparatus to stand over-night, although on the previous evening the gas was free from sulphuretted hydrogen, after twelve to twenty-four hours a reaction was obtained from the rubber. The use of a rubber balloon had therefore been discontinued. Dr. Rideal had used a carboy with a large gold-beater's skin balloon in it, the carboy holding about 40 litres, and, using the official apparatus with six jets, he had found the minimum delicacy to be one in five millions.

Mr. **HEHNER** said that the delicacy of the sulphuretted hydrogen test by lead paper was exceedingly great. It was unintelligible why sulphuretted hydrogen must on no account be present in coal-gas, while from 17 to 22 grains per 100 cubic feet of other sulphur compounds were permitted, seeing that the sulphuretted hydrogen would burn into exactly the same product as the other sulphur compounds. The nitro-prusside test, which was credited with being delicate, was a blunt test compared with the lead acetate test. *P*-amido-dimethylaniline gave a beautiful blue coloration (methylene blue) in the presence of sulphuretted hydrogen and ferric chloride, and had been recommended by Caro as a reagent for sulphuretted hydrogen. He (Mr. Hehner) had convinced himself that it was not nearly so delicate as lead acetate. Lead acetate solution was, again, much inferior to dry lead paper. Discoloration could be observed on the paper long before it was apparent in the solution. The preparation of mixtures of air or coal-gas with minute known amounts of sulphuretted hydrogen and the quantitative removal of the mixture from the vessel for the purpose of making the tests was by no means an easy one, as of course no substance or liquid could be used that might absorb the trace of sulphuretted hydrogen. While Mr. Dibdin had used an india-rubber balloon, by the expansion of which the gas was driven out, Dr. Rideal objected to the india-rubber on account of the difficulty with which the sulphuretted hydrogen that was absorbed by the balloon could again be driven out. Dr. Rideal had used gold-beater's skin instead. He (Mr. Hehner), however, found that the gold-beater's skin rapidly removed, probably on account of the oil with which the skin was prepared, every trace of sulphuretted hydrogen, both from air and from coal-gas, so that after a few hours' contact no H_2S was left. He had found it simplest to withdraw from a large vessel in which the gas mixture had been prepared by crushing calibrated capillary glass tubes filled with sulphuretted hydrogen, after allowing sufficient time for diffusion, some of the gas by the action of an aspirator or pump, measuring the gas thus withdrawn, under ordinary pressure. As to the general results, his experience was entirely in accordance with that of Mr. Dibdin.

Mr. GRIMWOOD, in reply, said that with regard to the question of the absorption of sulphuretted hydrogen by the india-rubber balloons and the subsequent giving of it up again, this has not been their experience.

When the apparatus was first put together they found that the rubber absorbed sulphuretted hydrogen, but after it had once had as much as it would take up, they had no further difficulty with it.

The mode of procedure certainly prevented the possibility of any occluded sulphuretted hydrogen affecting the result, as a blank determination was always made before any sulphuretted hydrogen was introduced into the apparatus.

With regard to the difference in sensibility found by Dr. Rideal and themselves, it must be remembered that two estimations of such difficulty as this cannot be expected to agree unless they are carried out under the same conditions, and it seems most probable that Mr. Hehner's experience of the absorption of sulphuretted hydrogen by the oil dressing of the gold-beater's skin, together with the difference in the two methods adopted, accounts for their want of agreement.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

Composition of Preserved Meats.—Part 10 of Bulletin 13 of the United States Department of Agriculture, recently issued, is devoted to the examination of preserved meats. Very many analyses have been made. Boric acid was found in many instances and in a few cases benzoic acid. These have been added probably as sodium salts. Small amounts of tin and zinc were found in many samples, tin being the more abundant. The amount was rarely over 200 milligrammes per kilogramme, and generally much less. No lead is reported.

The text of the bulletin gives account of the processes of analysis, most of which are standard methods, but some special methods are published. The dictum of some authorities that 1 per cent. of glycogen calculated to the dry, fat-free meat indicates horse-flesh is declared incorrect. The results obtained by examination of the fats are found to be better guides as to the source of the meats. The following table of constants will be, therefore, of interest.

Source of Fat.	Canned Roast-beef.	Canned Corned-beef.	Canned Smoked-beef.	Fresh Horse-meat.
Melting-point (in °C.) ...	36·5 to 43·9	37·2 to 43·4	37·7 to 43·1	27·2 to 32·5
Chilling-point (in °C.) ...	27·8 ,, 37·0	29·0 ,, 34·5	22·0 ,, 29·0	12·0 ,, 25·0
Iodine number	36·1 ,, 50·6	37·9 ,, 48·6	50·9 ,, 57·5	61·4 ,, 77·0
Maumené number	35·6 ,, 36·0	35·5 ,, 37·0	—	46·2 ,, 56·5
Degrees butyro-refractometer	47·0 ,, 55·5	52·7 ,, 56·0	51·0 ,, 58·5	55·2 ,, 76·5

The method for boric acid was as follows: Fifty grammes of the meat are incinerated (complete combustion is not essential), transferred to a short-necked flask, and acidified with hydrochloric acid. The flask is connected with a condenser, and four or five portions of 20 c.c. each of methyl alcohol are distilled by means of a calcium chloride bath into sodium hydroxide. The distillate is evaporated to dryness to expel the methyl alcohol, care being taken that it is distinctly alkaline; the residue is dissolved in 20 c.c. of water, acidified with hydrochloric acid, heated to the boiling-point to expel carbonic acid, and then titrated by Thompson's method. Each c.c. of decinormal alkali corresponds to 0·0062 of boric acid.

For the determination of saltpetre, which was found in small amount in a number of samples, several methods are given. One of these is termed the picric acid method, depending on the well-known reaction between the nitrates and phenolsulphonic acids, by which picric acid is formed, the amount being determined by colour comparison. The procedure recommended is as follows: 1 gramme of the sample is weighed into a 100 c.c. flask, 30 c.c. of water added, and heated on the water-bath for twenty minutes. Three c.c. of a saturated solution of silver sulphate are added for each per cent. sodium chloride present (previously ascertained), then 10 c.c. of lead subacetate solution and 5 c.c. of alumina cream, shaking after each addition. The mixture is made up to the mark with water, and filtered through a fluted filter, returning the liquid until it passes clear. Twenty-five c.c. of the filtrate are evaporated to dryness, 1 c.c. of phenolsulphonic acid added, and mixed well with a glass rod, then 1 c.c. of water and a few drops of strong sulphuric acid, and the mass heated for a few minutes on the steam-bath, care being taken not to char it. The mass is washed into a Nessler cylinder by means of about 25 c.c. of water, made alkaline with ammonium hydroxide, and made up to 100 c.c. The amount of nitrate is determined by comparing this colour with that produced by solutions containing known amounts of nitrates treated in the same way.

The Bulletin recommends the use of a reagent obtained by dissolving 2·5 grammes of phenol, 2·5 c.c. of water, and 55 grammes of sulphuric acid. This gives a mixture of ortho- and para-phenol, and has been shown to give somewhat uncertain colour effects. Better results are obtained by using the phenol disulphonic acid obtained by mixing 3 grammes of phenol with 37 grammes of sulphuric acid, and heating the mixture for six hours in boiling water. The reagent thus obtained forms picric acid in the cold with nitrates, and gives more constant colour effects.

H. L.

The Estimation of Sulphurous Acid in Dried Fruits. A. Beythien and P. Bohrisch. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1902, v., 401-409.)—The following quantities of sulphurous acid were found in various dried fruits (principally American), the results given being calculated into crystallized sodium sulphite ($Na_2SO_3 + 7H_2O$): Californian apricots, from 0·2162 to 1·1585 per cent.; Californian peaches, 0·9921 per cent.; Californian pears, 0·2399 per cent.; Italian prunes, 0·2637 per cent.; and "Görtzer" pears, 0·2901 per cent. The dried plums and apples examined were found to be free from sulphite. By cooking, less than one-half of the sulphite was removed from the fruits, and by soaking in water over-night and then cooking, a little more than one-half. W. P. S.

Estimation of Alkaloids in Cinchona Bark, etc. F. de Myttenaere. (Reprint from *Bull. Acad. roy. de médecine de Belg.*, *Séance* of January 25, 1902; through *Chem. Zeit. Rep.*, 1902, 118.)—The author has examined the methods which are official in the pharmacopœias of Austria (1893), Britain (1898), Germany (1900), Holland (1890), Italy (1892), and Switzerland (1893), as well as a process provisionally determined upon for the new Belgian volume; and he finds that they are all more or less faulty. He considers chloroform the only suitable solvent for the alkaloids, ammonia the best precipitant, and titration to be perfectly accurate. His own method is as follows: The sample is powdered till it will pass a sieve of 30 holes per linear centimetre; 7 grammes are then shaken at intervals for three hours in a 200 c.c. flask with 140 grammes of chloroform and 10 c.c. of 10 per cent. ammonia. Three grammes of gum tragacanth and 20 c.c. of water are next added, shaking thoroughly, and the vessel is rotated gently till the solid matter coheres. After standing one hour, 100 c.c. of the chloroform are quickly filtered off, the solvent removed by distillation, and the residue dried on the water-bath. It is taken up in the minimum of chloroform, poured into a separating funnel which contains 15 c.c. of decinormal hydrochloric acid, the basin rinsed twice with 5 c.c. of chloroform and a third time with a sufficient quantity of ether to make the organic solution float. The whole is well shaken for five minutes, the acid liquor run off through a wet paper, and the ethereal liquid washed three times with 10 c.c. of water. The filter-paper is also washed, and the excess of acid is finally titrated with decinormal sodium hydroxide, using hæmatoxylin as indicator. The process is applicable to the tinctures and extracts of bark; the dry extract is first dissolved in dilute alcohol, and the solution evaporated to dryness with powdered pumice. F. H. L.

ORGANIC ANALYSIS.

The Detection of Formaldehyde. C. Arnold and C. Mentzel. *Zeit. für Untersuch. der Nahr. und Genussmittel,* 1902, v., 353-356.)—For the detection of formaldehyde in meat, butter, milk, etc., from 5 to 10 grammes of the substance are shaken out with 10 c.c. of alcohol and the alcoholic extract is filtered. To 5 c.c. of the filtrate are added 0·03 gramme of phenylhydrazine hydrochloride, 4 drops of ferric chloride solution, and 10 drops of concentrated sulphuric acid, keeping the solution cool during the addition. Should formaldehyde be present, the solution is coloured red, whilst in the absence of this preservative it remains yellow. Light-coloured beers may be tested directly after adding a little alcohol, the red colour produced when formaldehyde is present being easily seen in the froth formed on shaking. The coloration may also be shaken out with ether, the latter being then drawn off and placed in a small flask. On adding a little alcohol and a drop of concentrated sulphuric acid, the red colour is reproduced. The above method will detect one part of formaldehyde in 10,000. W. P. S.

The Analytical Constants of Neat's-foot, Tallow, and Horse Oils. Augustus H. Gill and Allan W. Rowe. (*Journ. Amer. Chem. Soc.*, xxiv., 466.)—The authors have determined the various constants of a number of these oils, which are given in the following table :

	Specific Gravity, 15° C.	Valenta, 0° C.	Maumené, °C.	Specific Temp. Reaction.	Iodine No.	Titre Test, °C.	Iodine No. Fatty Acid.
Neat's-foot oil 1	0·915	70·0	42·2	87·9	72·9	19-20	68·6
,, ,, 2	0·914	75·5	42·2	87·9	72·9	18-19	64·6
,, ,, 3	0·919	51·0	49·5	103·1	67·1	17-18	67·3
,, ,, 4	0·916	61·5	42·2	87·9	72·1	16	69·5
,, ,, 5	0·916	75·5	42·2	87·9	66·0	25·5-26·5	63·6
	(100° C.)						
Tallow oil 1 ...	0·794	73·5	35·0	72·9	55·8	35-36	54·6
,, ,, 2 ...	0·794	71·0	35·0	72·9	56·6	36·5-37·5	57·0
,, ,, 3 ...	0·794	75·7	35·0	72·9	56·7	34·5-35·5	56·6

HORSE OILS.

Colour, etc.	Specific Gravity 15° C.	Valenta, 0° C.	Maumené, 0° C.	Specific Temp. Reaction.	Iodine No.	Iodine No. of Clear Oil.	Titre Test, 0° C.	Iodine No. Fatty Acids.
1. White-brown semi-fluid	0·919	80·2	46·0	95·8	75·1	—	32·5-33·5	72·9
2. Dark-brown semi-fluid	0·916	54·0	52·1	108·5	82·5	82·0	30·0-31·0	72·3
3. Yellow-brown liquid	0·922 (100° C.)	71·0	54·7	114·0	86·3	83·7	25·0-26·0	78·7
4. Golden-brown nearly solid	0·798	48·0	54·2	112·9	79·9	81·8	30-31	80·1
5. Very like 4	0·799	61·0	53·5	111·5	78·8	78·2	34-35	82·1

A. G. L.

A Test for the Gumming Quality of Lubricating Oils. Augustus H. Gill. (*Journ. Amer. Chem. Soc.*, xxiv., 467.)—The author has found that, in applying the elaïdin test to mineral oils, those oils which deposit the most "tar" change the most in use, and a relation appears to exist between the amount of change ("tar") and the oxygen absorbed. The gumming test was carried out by adding 11 grammes of Roth's liquid (nitro-sulphuric acid) to 5 grammes of the oil and stirring thoroughly, the mixture being kept at a temperature of 10° to 15° C. The oxygen absorbed was determined by weighing out 5 to 10 grammes of the oil into a flask, drawing out the neck of the flask into a long, thin-walled capillary, and after allowing the flask to cool to 20°, sealing off the neck. The flask was then heated for 100 hours at 80° to 105°, allowed to cool to 20°, the capillary broken under water, and the quantity of water which entered determined, giving the quantity of oxygen absorbed.

A. G. L.

Rubrescin, a New Indicator for Acid and Alkali Titration. A. Rosenfeld and J. Silber. (*Farmazeft*, 1902, x., 263; through *Chem. Zeit. Rep.*, 1902, 130.)—Rubrescin is prepared by heating together 50 grammes of resorcinol and 25 grammes of chloral hydrate in an oil-bath at 160° C., when hydrochloric acid is split off. The melt is a brittle non-hygroscopic mass, soluble in hot amyl alcohol, in cold methyl and ethyl alcohols, in water; slightly soluble in ether; insoluble in chloroform. It is purified by powdering it and extracting with chloroform. Made up as a 1 per cent. solution, rubrescin yields a dark-red liquid, 3 to 6 drops of which, with 1 drop of decinormal sodium hydroxide and 100 c.c. of water, give a red colour which is permanent for one hour, and remains as a red fluorescence after one day. The same effect is produced by 1 or 2 drops of decinormal borax and di- and mono-sodium carbonate. One drop of decinormal sulphuric acid completely destroys the red colour produced by 3 drops of the indicator; with 5 or 6 drops the liquid becomes yellow.

Ammonia strikes a red colour with rubrescin also. Experiments on the value of this new indicator in presence of carbon dioxide and for alkaloidal work are in progress.

F. H. L.

INORGANIC ANALYSIS.

Electrolytic Separation of Lead from a Phosphoric Acid Solution. A. F. Linn. (*Journ. Amer. Chem. Soc.*, xxiv., 435.)—The author shows that lead can be quantitatively separated by electrolysis from solutions of the phosphate in excess of phosphoric acid. The method pursued was to add to the lead solution, which should not contain more than 0·1 grammes of metal, 10 c.c. of sodium phosphate solution, and 12 to 14 c.c. of phosphoric acid (specific gravity 1·71), and then electrolyse for twelve to fourteen hours, using a current of $N.D._{100} = 0{\cdot}003$ ampère and voltage $= 3$. A smooth, uniform, gray coating is produced on the platinum dish which is washed first with water without interrupting the current, then very rapidly with a small quantity of absolute alcohol, and finally with ether, after which it is dried and weighed as metallic lead. If the current used is too strong, or if the electrolysis is continued too long, the deposit contains some phosphorus. If manganese is present in the solution some of it will be found with the lead, although manganese alone is not deposited from a solution of its phosphate in phosphoric acid.

A. G. L.

The Volumetric Determination of Zinc. E. Prothière. (*Journ. Pharm. Chim.*, 1902, xv., 419-422.)—The ordinary method of titrating the zinc with a standard solution of sodium sulphide suffers from the drawback that the lead acetate paper used as indicator reacts with the precipitated zinc sulphide.

Various attempts have been made to obviate this difficulty, but, according to the author, none of these are entirely satisfactory, and he therefore advocates the use of potassium antimony tartrate (tartar emetic) as an indicator in place of lead acetate.

Test paper impregnated with an aqueous 5 per cent. solution of tartar emetic gives no coloration until the whole of the zinc has been precipitated, when a yellow stain is instantly produced.

The test paper dried spontaneously in the air keeps well, and is very sensitive, giving a clear reaction with a solution of sodium sulphide containing 1 part in 10,000.

C. A. M.

Volumetric Determination of Iron. W. H. Gintl. (*Zeits. f. angew. Chem.*, 1902, xv., 398-402, 424-434.)—After discussing the various methods that have been proposed, the author describes experiments performed to investigate Storch's method, in which the iron solution is acidified with sulphuric acid, boiled for one and a half hours with metallic copper, and then titrated with permanganate. He found the results were always high in consequence of the formation of a small quantity of cuprous salt.

Excellent results were obtained by reducing with a spiral of palladium wire impregnated with hydrogen. The palladium may be charged either by making it the cathode of an electrolytic cell, or by heating it and allowing it to cool in a current of hydrogen. The solution, containing up to 0·4 gramme Fe_2O_3, is placed in a flask, 4 c.c. of sulphuric acid are added, the palladium spiral (about 3 grammes in weight) is introduced, the flask is closed with a Bunsen valve and heated on a water-bath for one and a half hours. The palladium is then removed, and the solution is titrated with permanganate. A. M.

The Volumetric Estimation of Alumina, and Free and Combined Sulphuric Acid in Alums. Alfred H. White. (*Journ. Amer. Chem. Soc.*, xxiv., 457.)—The author finds that if a solution of alum is titrated with barium hydrate solution in the presence of neutral potassium sodium tartrate, phenolphthalein being used as indicator, the quantity of barium hydrate required corresponds to the sulphuric acid combined with the alumina, plus the free sulphuric acid, the sulphuric acid present as alkaline sulphate not affecting the result. The tartrate is added to prevent precipitation of the alumina with consequent absorption of the pink colour. Precipitation of barium sulphate is also very considerably retarded. On the other hand, if sodium citrate is used instead of the tartrate, the barium hydrate required corresponds to the whole of the free acid, plus two-thirds of that combined with the alumina. The method is carried out as follows: 3 grammes of the alum are dissolved in 100 c.c. water. To 25 c.c. of this solution 50 c.c. of a neutral 10 per cent. potassium sodium tartrate solution are added; the solution is then titrated with barium hydroxide. Another 25 c.c. of the alum solution are evaporated to dryness on the water-bath; the residue is dissolved in 50 c.c. neutral 10 per cent. sodium citrate solution, allowed to stand for ten minutes, and titrated with barium hydroxide. The difference between the two titrations is equivalent to one-third of the sulphuric acid combined with alumina, and consequently to one-third of the alumina present. By multiplying this difference by three, and subtracting from the first value, the amount of free sulphuric acid is obtained. The barium hydroxide solution must be standardized against standard sulphuric acid solution in which approximately enough aluminium hydroxide has been dissolved to correspond to aluminium sulphate. The quantities of alumina found in this way are within 0·1 per cent. higher than those found gravimetrically.

The values for free acid found by this method are sometimes quite different from those found by the use of Beilstein and Grosset's method (*Bull. de l'Acad. Imp. des Sci.*, in St. Petersburg, 1890, 147), in which the free acid is extracted with alcohol after the addition of a large excess of ammonium sulphate to the saturated solution of the alum. It was found, however, that if the alum was directly dissolved in sodium citrate solution and the solution titrated at once, values similar to those given by this method were obtained. The explanation of these results lies in the fact that commercial alum is not always a homogeneous substance, often containing free acid in one part and basic salt in another. On solution, combination is only slowly effected. Beilstein and Grosset's method probably gives the amount of free acid present in the solid salt, but not the amount of free acid present in a solution of the salt which has been allowed to stand for some time. A. G. L.

The Determination of Lithia in Lepidolite. W. J. Schieffelin and W. R. Lamar. (*Journ. Amer. Chem. Soc.*, xxiv., 392.)—Decomposition of the mineral is effected by means of the J. Lawrence Smith method, using a low red heat for the fusion. The ammonium salts should be slowly driven off from the alkalies, otherwise lithium chloride may be volatilized. Lithium is separated from the other alkalies, after the last traces of calcium have been precipitated by ammonium oxalate, by the Gooch method as follows: The solution of the chlorides is filtered into a flask of 80 c.c. capacity and evaporated down to 1 or 2 c.c. A few drops of water and 1 or 2 drops of hydrochloric acid are added to effect complete solution, and then 15 c.c. of amyl alcohol. The solution is then carefully heated to boiling, and kept gently boiling until all the water has been driven off. At this point a thermometer held in the vapour should read 129° to 130°. The amyl alcohol when cold is decanted through a filter into a platinum dish, and the residue washed till free from lithia, about 20 c.c. amyl alcohol being used. The solution is then evaporated to dryness at 125°; 0·5 c.c. concentrated sulphuric acid is added to the residue, which is heated until the excess of acid has been volatilized, after which the sulphate is kept fused for one minute, covered, allowed to cool, weighed, again fused, and reweighed. The weight should be constant. The lithium sulphate obtained in this way is very nearly pure, rubidium and cæsium chlorides being practically insoluble in amyl alcohol. For the small amounts of sodium and potassium chlorides dissolved, the Gooch correction of 0·0011 gramme for each 10 c.c. amyl alcohol, exclusive of washings, is made. A. G. L.

The Presence of Barium in Mineral Waters containing Sulphates. P. Carles. (*Ann. de Chim. anal.*, 1902, vii., 91-93.)—The author has found that the waters of Néris-les-Bains contain lead, copper, barium, lithium, silico-fluorides, and borofluorides. As they were also found to contain sulphates and alkali bicarbonates, experiments were made to explain the simultaneous occurrence of barium and sulphuric acid.

In the neighbourhood of Néris several quarries of fluorides and one of the mineral barytes are being worked. Several grammes of powdered barytes were boiled for three hours with water containing pure sodium bicarbonate. The liquid was then found to contain undecomposed barium sulphate and silico-fluoride, the excess of sodium bicarbonate, and the products of their reaction—viz., barium carbonate, and sodium sulphate and silico-fluoride.

On now supersaturating the liquid (suitably diluted) with carbon dioxide, it became clear within twelve hours, whilst both barium and sulphates were identified in the filtrate.

From this the author concludes that mineral waters containing sulphates and bicarbonates are capable of decomposing barium sulphate if they contain an excess of free carbon dioxide, with the formation of soluble barium bicarbonate; and he considers it probable that the lead in the waters of Néris-les-Bains is due to a similar cause. C. A. M.

Preliminary Note on a New Separation of Thorium. Floyd J. Metzger. (*Journ. Amer. Chem. Soc.*, xxiv., 275.)—Thorium is stated to be quantitatively precipitated from 40 per cent. alcoholic solutions by means of fumaric acid, which produces no precipitate in cold solutions of cerium, lanthanum, or didymium. Consequently, thorium can be quantitatively separated from these elements by means of fumaric acid, a single reprecipitation sufficing to remove the traces of these substances which may be carried down in the first precipitation. A. G. L.

A New Method of separating Ceria from Mixtures of Rare Earths. R. J. Meyer and M. Koss. (*Berichte*, 1902, xxxv., 672-678.)—The following method is recommended by the authors for the rapid separation of cerium in a fairly pure condition. It is based upon the fact that cerium is precipitated from boiling solutions of ceric salts by magnesium acetate, whereas salts of lanthanum and didymium, etc., remain in solution.

In the case of a mixture of these earths the oxalates are converted into the compound ammonium nitrates, of which 100 grammes are dissolved in 2 litres of water in a large flask. The solution is heated to the boiling-point, and kept in motion by means of a current of steam, whilst a solution of 50 grammes of magnesium acetate in 500 c.c. of a 2·5 per cent. solution of hydrogen peroxide is introduced drop by drop, until a drop of the filtered liquid gives a pure white precipitate on treatment with ammonium hydroxide and hydrogen peroxide.

The liquid is then allowed to cool, and the orange-coloured precipitate of basic ceric acetate collected and washed with boiling water with the aid of a filter-pump, and dried at 120° C.

About 3 or 4 per cent. of didymium is simultaneously precipitated with the cerium salt, but the latter can readily be purified by dissolving it in nitric acid and converting it into ceric ammonium nitrate (*cf.* Wyrouboff and Verneuil, ANALYST, xxiii., 164). C. A. M.

Estimation of Silicon in Rich Ferro-Silicons by means of Peroxide of Sodium. C. Ramovino. (*Moniteur Scientifique*, xvi., January, 1902; through *Chem. News*, lxxxv., 218.)—Half a gramme of the finely powdered ferro-silicon is mixed with 10 grammes of a mixture of sodium and potassium carbonates and 1 gramme of sodium peroxide, and the whole slowly heated in a platinum crucible over the blowpipe. After decomposition is complete, the melt is allowed to cool, treated with boiling water and dilute hydrochloric acid in a porcelain dish, and the solution evaporated to dryness after adding 10 c.c. nitric acid and 2 grammes potassium chlorate. The residue is baked at 110°, taken up with 20 c.c. hydrochloric acid and 200 c.c. water, the solution boiled, filtered, and the insoluble residue ignited and weighed as silica. In the filtrate manganese and sulphur may be estimated. The method is especially applicable to those rich ferro-silicons which are not attacked by acids. A. G. L.

Note on the Purification of Hydrochloric Acid from Arsenic. L. T. Thorne and E. H. Jeffers. (*Proc. Chem. Soc.*, 1902, 118, [78].)—The hydrochloric acid to be purified is diluted with water until its specific gravity is 1·10, then raised to the boiling-point, and a piece of fine copper gauze introduced. The gauze should be of pure copper, of 100 meshes to the inch, and, for 2 litres of liquid, should be about four inches square, coiled very loosely round a long glass rod flattened at the end for convenience in handling. The whole is kept just boiling for one hour. If the gauze becomes blackened, it is replaced by a second, and, if necessary, a third piece. As soon as a piece of gauze remains bright after one hour's digestion, the acid is transferred to a retort without being allowed to cool, and distilled over another piece of gauze. As a precaution, the first 20 per cent. of the distillate are rejected, when 100 to 200 c.c. of arsenic-free acid are left in the retort. The method is simple, requires but little attention, and does not necessitate the use of the purest acid. The acid obtained is of constant boiling-point. A. G. L.

The Determination of Molybdic Acid after Reduction with Hydriodic Acid. F. A. Gooch and O. S. Pulman, jun. (*Zeits. Anorg. Chem.*, xxix., 353.)—From 0·3 to 0·5 gramme of the sample of molybdate, at least 40 c.c. hydrochloric acid, and 0·2 to 0·6 gramme potassium iodide are placed in a flask of 150 c.c. capacity, which is loosely closed by a bulb-tube (Fig. 1). The solution is boiled down to a volume of 25 c.c., which can be read off by means of a mark on the flask, immediately diluted to 125 c.c., cooled, and transferred to a Drechsel's wash-bottle fitted with a trap (Fig. 2) containing potassium iodide solution. About 0·5 gramme manganous sulphate is then introduced to prevent the production of chlorine during the subsequent oxidation. Deci-normal potassium permanganate solution is then run in from a burette until it is in excess, and the volume used noted. A quantity of standard arsenious oxide solution, roughly equivalent to the permanganate used, is then added, after which a solution of 3 grammes tartaric acid and a small excess of potassium bicarbonate solution are added. Finally, the glass stopper and tubes are rinsed into the bottle, the contents of the trap are added, and the excess of arsenious oxide is titrated with iodine solution.

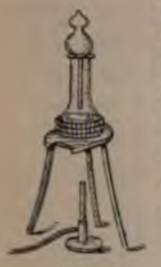


Fig 1.

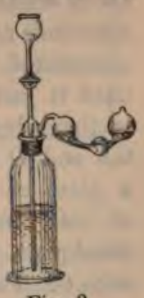


Fig. 2.

The amount of molybdic acid is given by the quantity of permanganate solution used plus that of the iodine solution, and minus that of the arsenious solution, one atom of oxygen being equivalent to two molecules MoO_3. The arsenious oxide is added to reduce the iodic acid, iodine, and higher oxides of manganese formed during the oxidation with permanganate. The oxidation can also be effected directly with iodine solution, but requires about two hours in that case, whereas with permanganate it is instantaneous. The test analyses given are satisfactory.

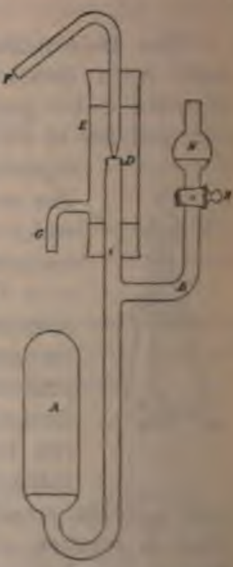
A. G. L.

Determination of Carbon in the Presence of Osmium. **G. v. Knorre.** (*Zeits. f. angew. Chem.*, 1902, xv., 393.)—When osmium is heated in the presence of oxygen it forms the volatile tetroxide—OsO_4—which is absorbed by potash solution just as carbon dioxide is. The gases can, however, be freed from osmium by passing them through a solution of ferrous sulphate. Acidified ferrous solution is placed in a flask, and the combustion gases are forced to bubble through it, after which they pass through an inverted condenser, then through a calcium chloride tube, and finally into potash bulbs. At the end of the operation the ferrous sulphate solution must, of course, be boiled.

A. M.

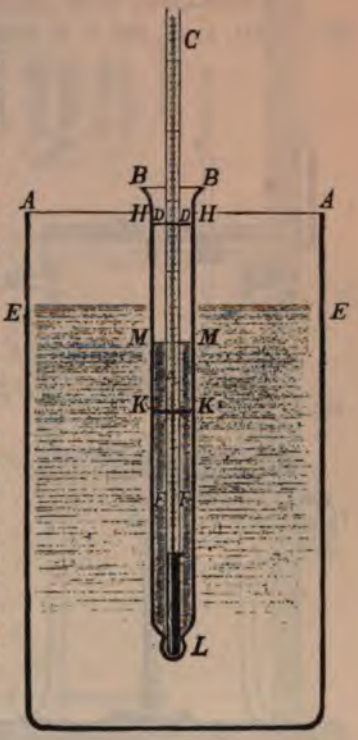
APPARATUS.

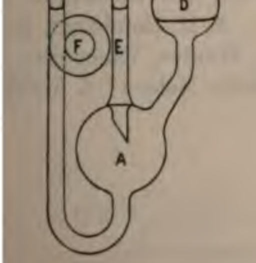
Modified Form of Thermo-Regulator. **T. S. Patterson.** (*Journ. Soc. Chem. Ind.*, 1902, xxi., 456.)—The bulb *A* is made from an ordinary test-tube, 6 inches by $\frac{3}{4}$ inch, and is filled, as in the ordinary forms, with toluene (air would probably do as well for most purposes), which is sealed by mercury, as usual. Some inches above the top of the bulb *A* the side-tube *B* is fixed. This tube extends vertically upwards, and is fitted with the stopcock *H*, just above which a bulb *K* is blown. The other portion *C* of the tube ends at the same level as the bulb *K*, where it is slightly constricted. Mercury is introduced into both limbs *B* and *C* until it just appears about to overflow at *D*. The stopcock is then closed, and a piece of thin sheet rubber laid over the end *D* so as to touch the mercury, and then tied with a piece of thread. A wide piece of glass tubing *E*, open at both ends and fitted with a short side-tube *G*, is then fitted on a rubber cork, previously placed in position on the tube *C*. The upper end of *E* is closed by another rubber cork, through which passes the gas inlet tube *F*. This latter is tapered, as shown in the figure, and the end cut off with a very slight slant. The tapered end of the tube is pushed through the upper cork until it reaches to within about $\frac{1}{2}$ a millimetre, or less, of the rubber membrane. The regulator is then ready for use. The gas-pipe is connected with *F*, and that to the burner with *G*. A small hole may be blown in *F* to act as a by-pass, or an external by-pass may be arranged if preferred. The temperature does not vary more than 0·01° in an hour. To adjust the apparatus, the bulb *A* is placed in the water-bath, the tap *H* opened, and the bunsen lit. The tap is closed when the water reaches a temperature 0·3° less than that actually required.



A. M.

A Melting-Point Apparatus. A. Klinkhardt.
n. Zeit., 1902, xxvi., 203.)—*B* is a glass cylinder
ated with mercury at the point *L*, and provided
a mark *H*. The thermometer, *C*, which is sus-
ed in any convenient way, bears a special mark *D*,
is graduated in fifths. *B* is filled to the level *K*
the liquefied substance whose melting-point has
ascertained, then fastened vertically in a con-
nt vessel containing ice-cold water, and the
nometer inserted so that the marks *D* and *H*
ide. When the material has solidified the
e apparatus is suspended inside a beaker con-
ng water at a temperature of 5° or 10° above
nelting-point expected. Directly melting begins
cur *B* falls slightly, and the marks *D* and *H*
ge; the apparatus is taken out of the beaker,
C is used as a stirrer until the mercury becomes
nary. The device is particularly suitable for
mining the liquefying points of solutions of
in and glue, which may be employed as 20 and
r cent. solutions respectively. F. H. L.

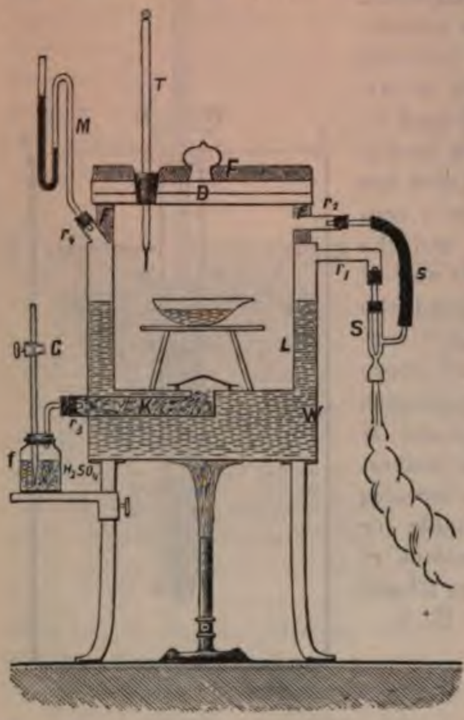




A New Design for Potash Bulbs. J. N. Tervet. (*Chem. News*, lxxxv., 112.)—The advantages claimed for the potash bulbs shown in the figure are that the potash is used up more uniformly than in the ordinary pattern, that crystallization of potassium carbonate does not occur, and that owing to the intimate mixture of gas and solution the speed of absorption is increased, and, consequently, the time required for combustion is diminished.

The apparatus may be obtained from Max Kaehler and Martini, Berlin. A. G. L.

A Water-Oven with constant Current of Dry Air. W. Gallenkamp. (*Chem. Zeit.*, 1902, xxvi., 249.)—It will be seen from the accompanying illustration that this oven is so constructed as to employ the steam raised in the jacket to work the injector *S*, whereby a steady current of air is extracted from the interior through the tube *s*. Air enters through the sulphuric acid bottle, the stream being regulated by the cock *G*; and in order to warm it before entering it is made to travel through the submerged tube *K* which is tightly packed with copper turnings. The cover is ground to fit, and carries a felt cover, *F*, to economize the heat. A pressure of 30 or 40 millimetres of mercury, indicated by the manometer *M*, suffices to work the injector. The apparatus may be fitted with a constant supply arrangement for the water; but in that case the inlet must be some 40 centimetres above the boiler level in order to establish the necessary pressure. A saline solution is advisable if a temperature of 100° is required. Liquids of higher boiling-points can also be employed in the jacket; if they are adopted, a condenser may be fitted to the outlet of the injector. The oven is made by the firm of Böhm and Wiedemann, of Munich.



F. H. L.

EXAMINATION UNDER THE BUREAU OF MINES AMENDMENT ACT OF BRITISH COLUMBIA, 1899.

The half-yearly examination for the qualification of Provincial Assayer, which was held at Victoria, British Columbia, on April 22, 1902, and the four subsequent days, resulted in the following gentlemen being granted licenses: A. B. Rombauer, Butte; G. Crearer, Boundary Falls; L. E. Gooding, Rossland; C. Hunter, Vancouver.

The examiners were: H. Carmichael, Victoria; H. Harris, Nelson; A. McKillop, Nelson; J. Cuthbert Welch, Trail.

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Editor: WALTER J. SYKES, M.D., D.P.H., F.I.C.

VOLUME XXVII.—No. 317.
AUGUST, 1902.

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THE ANALYST.

AUGUST, 1902.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

HEXABROMIDES OF GLYCERIDES AND FATTY ACIDS.

BY JOHN WALKER AND GEORGE WARBURTON.

(*Read at the Meeting, April* 9, 1902.)

IN a paper by Hehner and Mitchell, published in the ANALYST of December, 1898, there is given a convenient method for the determination of the hexabromides obtainable from oils and their fatty acids. This was accompanied by some figures obtained by the authors in the course of their investigations.

The following table may be of value to those interested in the subject, particularly as some of the figures given refer to oils not hitherto examined in this direction:

Kind of Oil.	Per Cent. Yield of Hexabromide from Glyceride of Fatty Acids.			Per Cent. Yield of Hexabromide from Fatty Acids.		
Japan fish	21·14	22·07		23·04		23·32
Newfoundland cod ...	32·68	30·62		39·1		37·76
Shark-liver	21·22	19·08		12·68		15·08
Genuine cod-liver ...	35·33	33·76		29·86		30·36
Deodorized fish	49·01	52·28		38·42		39·27
Sperm	2·61	2·42		2·05		—
,, ...	3·72	3·69	(stood 48 hours)	—		—
Whale	15·54	16·14		12·38		12·44
Seal	27·54	27·92		19·83		19·93
Rape	—	—		2·44		3·38
Linseed (iodine value 184)	—	—		31·31	30·44	30·80
Liquid acids (iodine value 208)	—	—		34·9		—
Linseed (iodine value 181)	23·14	23·52		29·06		29·34
Candle-nut	8·21	7·28		11·53	11·23	12·63
Chinese-wood (1st sample)	0·38	0·39		—		—
,, ,, (2nd sample)	none			—		—
Safflower	—	—		1·65		0·65

The work was carried out in the laboratory of Dr. Lewkowitsch.

DISCUSSION.

Mr. **Hehner** inquired whether Dr. Lewkowitsch (who read the paper in the absence of the authors) could explain why, in some cases, the fatty acids yielded so much smaller percentages of hexabromide than the glycerides.

Mr. C. A. **Mitchell** said that since the paper by Mr. Hehner and himself, to which reference had been made, he had examined a large number of linseed oils, and almost without exception the quantity of hexabromide yielded was from 23 to 25 per cent. In one case 42 per cent. had been obtained, but he did not know the origin of the oil. He had not been able to reduce the compound, even after treatment with zinc for several hours.

Dr. **Lewkowitsch** said that the point referred to by Mr. Hehner was a somewhat curious one, and required explanation. He was not himself responsible for the figures, which were simply given as they were obtained in the actual experiments. The collection, however, of individual figures such as these could not but tend to an increase of knowledge in regard to the individual oils concerned.

ON THE DETERMINATION OF MINERAL OIL IN ROSIN OIL.

By John H. Walker and Charles D. Robertshaw.

(*Read at the Meeting, April* 9, 1902.)

The correct determination of mineral oil in rosin oil is still an unsolved problem.

We recently had occasion to determine small quantities of mineral oil in rosin oil, and in the course of the examination we made a number of experiments with the methods which have been proposed for the solution of this problem. *McIlhiney's* method for estimating rosin oils in vegetable oils consists in ascertaining the bromine substitution figure. This is very low for vegetable oils, but somewhat considerable for rosin oils, the figures given by him for rosin oil varying from 42 to 90. Since the bromine substitution value of mineral oils must be very low, if any, it was thought that the application of this method might lead to good results. The first step was to ascertain whether the method yields constant results, and with this view a genuine rosin oil was examined, with the following results:

Bromine Values of Rosin Oil.

	Bromine Absorption.	Substitution.	Addition.
No. 1 ...	112	58·37	− 4·34
No. 2 ...	116·5	59·95	− 3·40
No. 3 ...	105·8	56·56	− 7·32

A second set of experiments led to absorption figures varying from 95·7 to 129·2. Another genuine rosin oil gave values varying from 77·9 to 109·1.

This proves that the reaction taking place is not a very definite one, and that the results depend not only on variations of time, temperature, and other conditions, but even in comparative tests and under exactly the same conditions the results remained very unsatisfactory. In fact the numbers obtained above were got in experiments worked under exactly identical conditions, and using ice, as directed by McIlhiney. This is further emphasized by the very erratic figures obtained when applying the Hübl or Wijs methods to the oils in question.

Similar tests made with two mineral oils led to the following figures:

	Bromine Absorption.	Bromine Value calculated from Iodine Value.
Mineral oil, No. 1 ...	16·8 to 29·2	9·6
,, ,, No. 2 ...	21·9 to 27·4	11·7

From the foregoing it would appear a hopeless task to determine quantitatively the rosin oil in a mixture of rosin oil with mineral oil. This is shown in several experiments carried out by mixing 90 parts of rosin oil No. 1 with 10 per cent. of mineral oil No. 1. The figures obtained were as follows:

	Bromine Absorption.	Bromine Value calculated from Iodine Value.
Mixture of 90 per cent. rosin oil No. 1 and 10 per cent. mineral oil No. 1	70·4 to 98·6	33·8

Recently *Holde* proposed a method for detecting and determining rosin oil and mineral oils by their different solubility in 96 per cent. alcohol, when 10 c.c. of the sample are shaken with 90 c.c. of 96 per cent. alcohol in a stoppered measuring cylinder, the refractive index of the insoluble portion being then taken. The undissolved portion would indicate the presence of mineral oil. This naturally presupposes that pure rosin oil is completely soluble, and that may have been the case with the rosin oils examined by Holde, but the samples of pure rosin oils which we tested left a considerable residue at the bottom of the cylinder. In the case of rosin oil No. 1 about thirty per cent. was left undissolved, and in the case of rosin oil No. 2 forty per cent. Whether this is due to our rosin oils having been freed from the rosin acids or not has not been investigated, but at any rate we feel justified in concluding that Holde's method does not admit of general application. The sample of mixed rosin and mineral oils—90 and 10 respectively—gave 60 per cent. of insoluble substance.

The best method of determining the mineral oil in rosin oil was found to be Valenta's acetic acid method. The percentage of rosin oil dissolved in the acetic acid was, in the two cases of genuine rosin oils, 16·8 and 16·6 per cent. respectively, which compares well with Valenta's figure of 16·9 per cent.

The mineral oil No. 1, used for preparing the mixture, gave 4·4 per cent. (Valenta stating 2·6 to 6·5 per cent.). A mixture prepared from equal parts of the mineral oil and the rosin oil No. 1 gave 10·84 per cent. insoluble, theory requiring 10·59 per cent.

So far, then, our experience shows that Valenta's method is the best. As a preliminary test, the determination of the specific gravity will certainly give very valuable hints.

The above experiments were carried out in the laboratory of Dr. J. Lewkowitsch.

DISCUSSION.

Mr. **Jenkins** said that of the mineral oils, that from Borneo was the one which came nearest in its reactions to rosin oil. Its specific gravity was from 0·97 to 0·99, and it was very readily soluble in an equal volume of acetone. It did not give the Liebermann-Storch reaction, but in several other respects, and especially in solubility, it approximated to rosin oil.

Dr. **Lewkowitsch** observed that the peculiarities of Borneo oil afforded a further proof of the danger of generalizing from the results yielded by a limited number of samples. If this short paper were merely considered as a sort of warning, he thought the authors would have done well in drawing attention to the matter.

THE COMPOSITION OF MILK.

By H. Droop Richmond, F.I.C.

(*Read at the Meeting, May* 7, 1902.)

Mean Composition during 1901.—Of the 37,662 samples examined in the Aylesbury Dairy Company's Laboratory during 1901, 32,635 were analyses of milk. The composition of 13,936 received from the farms is given in Table I.

TABLE I.

Average Composition of Milk during 1901.

Month.	Morning Milk.				Evening Milk.				Average.			
	Specific Gravity.	Total Solids.	Fat.	Solids-not-Fat.	Specific Gravity.	Total Solids.	Fat.	Solids-not-Fat.	Specific Gravity.	Total Solids.	Fat.	Solids-not-Fat.
January ...	1·0328	12·58	3·52	9·06	1·0327	12·90	3·83	9·07	1·0328	12·73	3·67	9·06
February...	1·0328	12·58	3·54	9·04	1·0326	12·94	3·88	9·06	1·0327	12·74	3·70	9·04
March ...	1·0326	12·49	3·50	8·99	1·0325	12·85	3·85	9·00	1·0325	12·65	3·66	8·99
April ...	1·0323	12·38	3·46	8·92	1·0320	12·73	3·85	8·88	1·0321	12·55	3·66	8·89
May ...	1·0326	12·25	3·29	8·96	1·0323	12·70	3·76	8·94	1·0324	12·47	3·52	8·95
June ...	1·0326	12·25	3·31	8·94	1·0320	12·58	3·70	8·88	1·0323	12·42	3·51	8·91
July ...	1·0319	12·17	3·41	8·76	1·0313	12·37	3·69	8·68	1·0316	12·27	3·55	8·72
August ...	1·0319	12·27	3·47	8·80	1·0313	12·59	3·85	8·74	1·0316	12·43	3·66	8·77
September	1·0321	12·47	3·59	8·88	1·0317	12·89	4·03	8·86	1·0319	12·68	3·81	8·87
October ...	1·0321	12·58	3·69	8·89	1·0316	13·02	4·15	8·87	1·0318	12·80	3·92	8·88
November	1·0321	12·74	3·82	8·92	1·0317	13·03	4·14	8·89	1·0319	12·89	3·98	8·91
December	1·0322	12·75	3·80	8·95	1·0319	13·13	4·18	8·95	1·0320	12·94	3·99	8·95
Average	1·0323	12·46	3·53	8·93	1·0320	12·81	3·91	8·90	1·0321	12·63	3·72	8·91

The morning and evening milks have been kept separate, and show the usual average difference of 0·3 to 0·4, which has been always previously observed. In previous years the average percentage of fat has usually shown a slight fall from that found in the previous year; in 1901 there is a distinct rise, the percentage being 3·72 as against 3·64 in 1900. This rise is chiefly due to higher figures in the last four months, which average 0·16 per cent. higher than the corresponding months of 1900, the rise in the previous eight months averaging only 0·03 per cent.

The cause of this is uncertain, but the fact that the rise in fat percentage is coincident with the establishment of an official standard may indicate that milk-producers are paying more attention to the quality of the milk yielded than formerly.

The former experience that the lowest fat occurs in May and June and the highest in the winter months, and that the solids-not-fat are low in July and August, is again repeated.

Accuracy of Methods Used.—As in the bulk of the analyses the fat is determined by the Gerber method, I have compared the results with those obtained by the Adams method for fat.

In 52 analyses the difference between Adams and Gerber was:

In 40 cases, or 76·9 per cent., 0·1 or less;
In 11 cases, or 21·2 per cent., 0·1 to 0·15; and
In 1 case, or 1·9 per cent., above 0·15.

The mean Gerber figure was 0·006 per cent. higher than the mean Adams figure.

The Proteids of Milk.—In a paper read last year (ANALYST, xxvi., 310) I showed that when the minimum quantity of a mineral acid was added to milk the quantity necessary to just curdle the milk on boiling was practically equal to the amount necessary to replace the sodium by hydrogen in the formula:

$$C_{162}H_{255}N_{41}SPO_{52}CaNa.\tfrac{1}{2}(Ca_3PO_4).$$

As a further confirmation of the view that this is the action that takes place, I determined the casein, ash, lime, and phosphoric acid in a sample of milk, and in the serum obtained by curdling with a minimum quantity of hydrochloric acid at the boiling temperature the figures were:

	Milk.	Serum.	Difference.
Casein	3·05	—	—
Ash	0·79	0·56	0·23
CaO	0·176	0·056	0·120
P_2O_5	0·232	0·112	0·120

The figures calculated for the removal of Ca and $\tfrac{1}{2}(Ca_3PO_4)$ with each molecule of casein are:

Ash	0·234
CaO	0·116
P_2O_5	0·118

The difference between the solids-not-fat after allowance for the ash was 2·91 per cent., and I have constantly found that the quantity of casein removed does not quite coincide with the estimate of the quantity of casein present. I do not propose at present to discuss the significance of this fact.

In the same paper I also mentioned that on filtration through porcelain the casein and albumin were both removed. There is a possibility that both albuminoids may exist in milk in combination, and the compound may only be split up under the influence of reagents. It appeared to me that filtration through filter-paper would settle the question. I filtered half a litre of milk kept at 0° C.; three fractions were collected, and it was found that the last fraction, which contained a trace of casein, contained sensibly the same amount of albumin as the portion left on the filter, which was very rich in casein. As it does not appear probable that filtration through paper would break up a compound, it may be concluded that casein and albumin are not decomposition-products of one albuminoid existing in milk.

Attempts were made to ascertain whether any change in the acidity of milk took place when the albumin was coagulated by boiling; a quantity of acid sufficient to curdle the milk at temperatures below the coagulating point of albumin was added to milk, the serum removed, and the acidity estimated before and after boiling. The results showed considerable variation, the reduction of acidity varying from 3·6° to 5·3° in the same milk; it was, however, found impossible to completely remove the casein, which probably existed in suspension, and was removed on boiling. The casein was estimated as organic phosphoric acid by the method given in "Dairy Chemistry" (p. 113),* and the greatest reduction was found where the greatest amount of casein was present, and *vice versâ*. The amounts of casein found would account for the greater portion of the reduction of acidity, and that due to the coagulation of the albumin must be extremely small; as experimental error would bear a large proportion to the absolute amount, it appeared hopeless to attempt quantitative determinations in this way.

A series of experiments was carried out to ascertain the strength of casein as an acid. It was certainly stronger than carbonic acid, as $\frac{1}{100}$ N. solutions of sodium and potassium carbonate dissolved 1·86 and 1·83 per cent. of casein respectively, the quantity calculated for the replacement of 2H by the alkali in $C_{162}H_{258}N_{41}SPO_{52}$ being 1·84. Boric acid produced in concentrated solution a partial coagulation on boiling, and casein is probably a somewhat stronger acid than boric under these conditions. Organic acids, such as acetic and lactic acids, were distinctly stronger than casein, as nearly the same amounts (9 to 10 c.c. normal acid per litre) were required to curdle milk as those of mineral acids (8·6 c.c.). Phosphoric acid was quite anomalous in its behaviour, as about 34 to 35 c.c. were required to curdle milk on boiling. This behaviour of phosphoric acid incidentally shows that milk is neutral to the casein compound which exists in milk, as, were it not so, a mineral acid should liberate phosphoric acid from the phosphates present, and a larger quantity of acid than that required to replace one atom of base for each molecule of casein should be necessary. This, I think, indicates that casein is an acid of the same strength as the second hydroxyl of phosphoric acid, as I have previously shown (*loc. cit.*) that the phosphates of milk are mono- and diphosphates.

Composition of Human Milk.—Four samples of human milk were examined. The composition was:

	I.	II.	III.	IV.
Specific gravity	1·0329	1·0294	1·0285	—
Total solids	11·45	10·48	11·90	13·77
Fat	2·53	2·63	4·00	5·60
Sugar (by difference)	6·87	6·34	6·06	6·74
Proteids	1·77	1·25	1·62	1·24
Ash	0·28	0·26	0·22	0·19
Zeiss refraction of fat at 35° C.	51·5°	—	—	53·2°
Sugar (gravimetric as lactose)	6·42	—	—	—
,, (polarized as lactose)	—	5·62	—	—
,, (polarized as sugar, having $[\alpha]_D$ 48·7°)	—	6·39	—	—

---

* I now find that it is unnecessary to add alkali to the filtrate from the mercuric nitrate solution before evaporation and ignition; the ash is always strongly alkaline without this.

The only figures calling for any remark are the sugar estimations. In conjunction with Professor Carter (*British Medical Journal*, 1898, No. 1) I have shown that the sugar of human milk appeared to differ from lactose, and had an $[\alpha]_D$ for the anhydrous sugar of 48·7°; the cupric reducing power is also slightly less than that of lactose. The present figures bear this out.

NOTE ON A CONVENIENT APPARATUS FOR THE CHEMICAL AND BACTERIOLOGICAL EXAMINATION OF THE ATMOSPHERE.

BY WILLIAM CHATTAWAY, F.I.C., AND F. M. WHARTON, F.I.C.

(*Read at the Meeting, May* 7, 1902.)

As impurities in the air, or at least such gases as have to be estimated in the ordinary way in the atmosphere, are present in comparatively small quantities, and as the error of experiment naturally affects such determinations in proportion to the volume of air examined, we have thought it best to adopt a plan whereby a very large volume of air may be tested, instead of using a few litres as has usually been done previously.

Some years ago one of us had occasion to examine large volumes of ozonized air, and the accuracy of the anemometer as a means of measurement was clearly established. Indeed, it was partly owing to the experiments referred to that the present apparatus was made.

We have recently had occasion to examine the atmosphere in and around a large chemical works where smelting processes were conducted, and the satisfactory way in which the apparatus worked seemed to justify the matter being brought to the notice of this Society.

In order to bring whatever liquid is used to fix the impurities sought for into contact with the air to be examined, we are satisfied that a jet apparatus is the most efficient and, at the same time, the most expeditious. For example, it is possible, in the course of half an hour, to treat at least 100 cubic feet of air, and, with the exception of CO_2 (which is admittedly somewhat difficult to determine by this method on account of the slowness of absorption), we believe that almost all other impurities can be dealt with in an exceedingly efficient and expeditious manner.

Briefly, the apparatus consists of a pump capable of giving a pressure equal to about 2 inches of water, which is used to force the air under examination through the perpendicular tube of an ordinary atomizing jet. The jet is placed inside a fairly large carboy, and the exit for the scrubbed air is an essential feature. This consists of a little spray trap, which is always kept wet during the process. The air is forced through the carboy by means of pressure from the pump, and the volume passed is finally measured by the anemometer. The anemometer must be protected against any form of back-pressure. The size of the carboy, the jet, and, within certain limits, the pressure, may be varied to suit any particular purpose, but we much prefer to work on a reasonably large scale.

Satisfactory results can be obtained with a powerful foot-pump, provided the metal parts are covered so as to guard against contact of the moist air with the metal, but, wherever possible, we prefer to use a pump similar to the one we have here to-night, driven by some convenient power. The chief points about this pump are that it has a flexible piston, that it does not require any form of lubrication, and that the few metal parts present are coated with anti-sulphuric enamel. This renders the likelihood of chemical action quite out of the question.

We do not claim that this apparatus is novel, except in so far as the use of an anemometer is concerned and the treatment of large volumes of air, but we do claim that it is a convenient and very reliable means for determining the impurities sought for.

We find that the spray method has been previously employed, and it is doubtless a very old plan, but we are not aware that it has been used either for bacteriological purposes or for dealing with large volumes of air as we suggest.

The only fault that appears to exist in the use of the apparatus is the inconvenience of the liquid being carried forward during the experiment. The accuracy, however, which is obtained by working on such large volumes more than compensates for the trouble which is thus involved.

With regard to special estimations, we may mention that we have not established that CO_2 can be readily estimated by one scrubbing, but it is not important, as one can easily obviate the errors arising from CO_2 when estimations of other gases are made.

We have obtained some encouraging results on the determination of SO_2 as $BaSO_4$ after oxidation of the aqueous liquid, but further experiments are being made in this direction.

Metallic impurities, such as lead, zinc, etc., in the form of dust or fumes, flue, gases and the like, can be readily dealt with by means of dilute alkali. If, however, soda is used it should be prepared from metallic sodium. Dust can also be readily removed by merely scrubbing with water.

Ozone can be readily estimated by the usual methods, and nitric oxide, free and albuminoid ammonia can be likewise estimated by this method.

In addition, the speed which may be obtained on a large scale is such as to justify, in our opinion, the possible application of this method for removing dust particles from the air in the case of breweries, photographic works, etc.

On a much smaller scale, but adopting exactly the same principle, an apparatus is shown which may be readily and quickly used for bacteriological examination of the atmosphere. In this case the only difference is that the volume of air is much smaller, and therefore need not be measured by means of an anemometer; but the air which has been forced through the jet can be measured by collection in a graduated vessel.

We prefer to directly scrub the air with sterile nutrient broth, precaution being first taken to thoroughly sterilise the apparatus. In order to obviate possible risk of contamination, if the broth were removed in the ordinary way, a little side-opening is made which will permit of a sterile pipette being used for removing the broth. In this way it is quite easy to determine, from a known volume of air and using a known volume of broth, the number and nature of the organisms found. We suggest that this method is particularly applicable to examination of the atmosphere of breweries and similar works where the introduction of foreign micro-organisms is of vital importance. Of course, instead of using nutrient broth, it is quite possible to use sterile wort or other suitable liquid media.

We are aware that the same results are obtained with other forms of apparatus, but we do not think that the same degree of accuracy has been obtained. We have established that it is possible to remove all the micro-organisms in the atmosphere with this apparatus. In the case of the Hesse apparatus, we think there is a certain risk of not removing all the micro-organisms; and, again, with Hueppe's method, where the air is bubbled through warm nutrient gelatin, we think that objection can be taken as to the thoroughness of this operation compared with that of the atomizing method which we suggest.

THE EXAMINATION OF WATER FROM SOME TYPHOID-POLLUTED WELLS.

By Samuel Rideal, D.Sc., F.I.C.

(Read at the Meeting, June 4, 1902.)

In December last year I received two samples of water from shallow wells for partial analysis as to whether they were suitable for drinking purposes. The chemical results for the two waters were as follows:

Parts per 100,000.

	No. 222.	No. 223.
Total solids	29·36	75·92
Chlorine	1·48	3·96
Nitric nitrogen	2·59	2·54
Nitrous nitrogen	Nil.	Trace.
Free ammonia	0·0045	0·0028
Albuminoid ammonia	0·0075	0·0132

Having analysed a considerable number of waters during the last few years from the shallow wells in this district, I have been able to fix a fairly rigid standard, from which I did not hesitate in condemning No. 223 as badly polluted and No. 222 also as unsuitable for drinking purposes, mainly on account of its high nitric nitrogen figure. After the analyses, I received notification from the sanitary inspector to the effect that both the waters had been taken where there was a case of typhoid fever in the house—No. 222 from a cistern in the house fed from a well in the grounds, depth unknown; and No. 223 from a garden well-pump about 20 feet deep—and a letter from the medical officer remarking on the somewhat high standard which I adopted for these shallow well waters. On receiving this information, although both the samples had been collected in ordinary stoppered half Winchester quart bottles not previously sterilized, and had stood about the laboratory some days, I determined to proceed with a bacteriological examination of the residues, amounting in each case to about 250 c.c., and containing the sediment from the whole of the sample.

From the residue of No. 223, the water showing the worse chemical results, no coli or other suspicious organisms were obtained, and, in fact, the bacteria growing at blood-heat seemed to have entirely disappeared. From the residue of No. 222 a number of more or less typical cultures of *Bacillus coli* were obtained, and an organism which, from its growth and reaction with the following different cultural tests, was almost certainly *Bacillus typhosus*:

Surface colony on agar, closely resembling typhosus; delicate, light-brown colour, finely granulated, and no centre.

Gelatin streak culture; grew somewhat slowly at 20° C., and no signs of liquefying after some weeks' incubation.

Agar streak culture; a marked growth in twenty hours at 37° C., resembling typhosus.

Culture in peptone water at 37° C.; growth, but no indol reaction could be obtained after four days' incubation.

Milk culture at 37° C.; not coagulated after seven days' incubation.

Agar shake culture at 37° C.; no gas formation.

Potato at 37° C.; no visible growth after twenty hours.

Proskauer and Capaldi's media; twenty hours at 37° C.—

> "No. 1." No production of acidity or apparent growth (resembling typhosus; coli organisms grow, producing marked acidity).
>
> "No. 2." Growth, but no acid reaction.

In all these tests it will be seen that, with the solitary exception of its failure to produce acidity in twenty hours in Proskauer and Capaldi's No. 2 medium, it conforms to the typhoid bacillus. Examined microscopically, the organism was seen as very motile short rods, with occasional long threads bent at an obtuse angle. Stained by Van Ermengem's process, flagella were demonstrated, but they were not so numerous as in other typhoid cultures, and in other respects the organism was proved to be enfeebled and not so resistant to the action of chemicals as stock cultures of *Bacillus typhosus*.

A report of this was sent to the medical officer, who very kindly forwarded some of the typhoid patients' blood for Widal's agglutination test. A negative was obtained with normal blood serum, and very rapid and marked clumping with the anti-typhoid blood serum from the patient.

DISCUSSION.

The PRESIDENT (Dr. VOELCKER) observed that the main ground for suspicion in the case of the first water was the large amount of nitrates present. It would have been interesting to know the amount of oxidizable matter in this water, and whether there was anything in the appearance of the sample that would arouse any suspicion of its bad quality. Certainly the water would seem, on the chemical figures, to be one to which some suspicion might attach. Still, the strictly chemical data available were perhaps scarcely sufficient to decide upon alone. If, however, associated with the amount of nitrates present, there was a considerable amount of organic matter, or if there was anything in the appearance of the water that would lead one to think that it was not altogether right, there would be confirmation of the suspicion. The second water, presumably, was also from a shallow well, and quite near the first one. Still, the great variation in the amount of total solids required some explanation. It might naturally be asked, too, whether, as in the case of the first water, confirmatory indications of bad quality were yielded by bacteriological examination.

Mr. W. H. JOLLYMAN inquired whether *Bacillus coli* had been isolated from these waters, as well as the supposed typhoid organism, and also whether any experiments had been made to ascertain if, in the case of the patient referred to, the blood clumped the *B. coli,* and if the organism in question was clumped by normal blood. He thought that such control experiments would be necessary before one could say for certain whether or not the organism was typhoid.

Dr. RIDEAL said that he had experimented with normal blood, and had found that the organism did not agglutinate with it. He had not tested the patient's blood with *B. coli*, because he was certain from his previous examination that this particular organism was not *B. coli.* All the tests proved that it was not *B. coli*, and pointed to its being typhoid, and he regarded the agglutination test as confirming what might be called the negative results obtained in the previous work. *B. coli* had been isolated from, and was present to a considerable extent in, sample No. 222; but in the case of sample No. 223, the organisms developing at blood-heat had apparently disappeared during the time the sample had remained standing. In the unpolluted shallow well-water of the district the chlorine was always much lower than was the case in these samples, as was also the nitric nitrogen. He should regard 1 part in 100,000 as a fair normal figure for the chlorine, and less than 0·6 part for the nitric nitrogen. He was unable to state the amount of oxygen absorbed by oxidizable organic matter in either of these two waters, but the opinion he had formed without the aid of that factor had been shown, he thought, to have been a justifiable one.

ON BELFIELD'S TEST FOR BEEF-STEARINE IN LARD.

By OTTO HEHNER.

UPON the form of the crystalline deposit obtained from an ether solution of lard is based the only method extant for the detection of beef-fat in lard. Pure lard, as a rule, gives, as is well-known, crystals which are flat, with chisel-shaped ends, sometimes single, often bunched together, and radiating from a centre, while in presence of beef-fat the crystals form exceedingly thin needles, curved, united in the form of wheatsheaves or horsetails, with pointed ends.

It has been shown by Mitchell and myself (ANALYST, vol. xxi., p. 328, and *American Chem. Soc. Journal*, vol. xix., 1897) that the "form of the beef-crystals is solely due to a larger proportion of stearic acid than can be obtained from pure lard by a single crystallization," and that by recrystallizing the deposit the stearic acid accumulates to such an extent, even in deposits from pure flare lard of average composition, that the crystals become indistinguishable from those obtained from beef-fat.

In the paper referred to a number of determinations of stearic acid in lards and other fats are given, showing that the highest percentage of stearic acid met with by us up to that date was 16. It was to be expected that in lards with a higher proportion of stearic acid the form of the crystals would more and more nearly approach to that associated with beef-fat. I have lately melted out in the laboratory from a piece of pig's flare some lard which gave an iodine absorption of only 45·6, and containing, as the average of two satisfactorily agreeing determinations, no less than 24·91 per cent. of stearic acid. The crystals from this sample are indistinguishable from those of lard largely admixed with beef-fat. In a second sample of flare-fat I found 20·75 per cent. of stearic acid; the ether-deposit from this sample also closely resembled that of beef-stiffened lard. A sample of mutton-fat contained 27·4 per cent. of stearic acid, and the crystals had all the appearance of beef-stearine crystals.

As, with the exception of the figures published by Mitchell and myself, no data as to the percentage of stearic acid in fats, based upon direct determination, are available, I think it worth while to place the above numbers on record. They should make analysts very cautious in placing reliance upon the form of the ether-deposits. When cotton-seed or other vegetable oil can be traced, and the iodine absorption is high, the "beef-form" of crystals may be taken to afford evidence of the presence of beef or mutton fats. In the absence of vegetable oil and with a low iodine absorption, the Belfield test can at most afford some reason for suspicion, but no trustworthy evidence of adulteration.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

The Analysis of Dutch Butter. **A. Kickton.** (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1902, v., 458, 459.)—Attention is drawn to the desirability of applying the phytosterol acetate test (ANALYST, xxvii., p. 94) to Dutch butters which give a low Reichert-Meissl number, and with which the colour tests for cotton and sesame oils yield negative results, before giving a definite opinion as to the presence of margarine in the butter. W. P. S.

The Polarization of Fruits, Jellies, Jams, and Honey. **L. M. Zolman.** (*Journ. Amer. Chem. Soc.*, xxiv., 515.)—From his experiments the author comes to the conclusion that the presence of hydrochloric acid increases the lævo-rotation of an invert sugar solution, and that this explains why, when hydrochloric acid is added to a solution of sucrose inverted by boiling with an organic acid, such as occurs in jams and jellies, a slightly higher reading is always obtained. The increase is proportional to the quantity of hydrochloric acid present, and to the amount of polarization, at any definite temperature. Introducing the correction calculated from his results into the Herzfeld formula, he obtains

$$S = \frac{a - b}{141{\cdot}85 + 0{\cdot}05b - \frac{T}{2}}$$

where S = sucrose, a = direct reading, b = invert reading, T = temperature. The modified formula corresponding to the method used by the Association of Official Agricultural Chemists is

$$S = \frac{a - b}{141{\cdot}79 + 0{\cdot}062b - \frac{T}{2}}$$

whilst, to calculate a small amount of cane-sugar in the presence of a large amount of invert sugar, the formula

$$S = \frac{a - [b - (0{\cdot}062 \times b)]}{141{\cdot}79 - \frac{T}{2}}$$

should be used.

The presence of hydrochloric acid has no effect on the polarization of solutions of glucose.

A. G. L.

Valuation of Flour for Bakers' Use. A. Maurizio. (*Landw. Jarhb.*, 1902, xxxi., 179; through *Chem. Zeit. Rep.*, 1902, 155.)—The author's experiments show that no opinion as to the suitability of a flour for baking can be based upon an extraction of the gluten, followed by an estimation of its power of rising when tested in an aleurometer, baking oven, or oil-bath. In general, however, the volume of the bread increases as the volume of the gluten. The quality of the gluten is an important factor; but, although its valuation, according to the usual practical methods, throws some light upon the quality of the flour for baking, the desirable qualities of the gluten cannot be formulated in precise language. The chemical processes of Robine and Girard are useless. Neither the amount of extractive matter recovered from gluten or flour, nor the specific gravity of solutions in acetic acid and alcoholic potash indicates the value of a sample to the baker. An estimation of the bulk of the bread or of the experimental cakes made in sheet-metal vessels with maize grits, glass pearls, etc., is of no value because of the different conditions in which they are obtained and of their irregular shapes. The volume of the fermented dough and of the finished bread do not depend upon the absolute quantity of carbon dioxide liberated by the yeast, but upon the power of the dough to retain more or less gas before it sets. Poor flours lose gas during fermentation; better descriptions hold it until they are placed in the oven. It would be advantageous if the loss of gas before baking could be avoided; but, on the other hand, there is a maximum volume for the bread, above which its quality suffers. The fermented doughs of different flours show differences in volume; but those differences are smaller than such as are given by different sample cakes of the same flour, while the volumes of the doughs do not agree with the volumes of the finished cakes. Fermentation tests, accordingly, cannot replace baking tests. The specific gravity of the bread is an excellent criterion for judging of the value of a flour or wheat for bakers' use. Products of the best quality have a specific gravity of 0·23 to 0·25; medium qualities show 0·35; inferior grades give 0·46 or more.

F. H. L.

On Specific Sera for Human Blood. Linossier and Lemoine. (*Acad. de Médecine*, March 25, 1902; *Ann. de Chim. anal.*, 1902, vii., 185.)—The authors have found that the specific character of the sera obtained by injecting rabbits with human serum (ANALYST, xxvii., p. 194) is not so absolute as has frequently been asserted. According to the results of their experiments the sera are not specific when a too concentrated solution of blood serum is employed, although the precipitate is more pronounced than in the case of the blood of other animals.

To avoid this source of error the test should only be applied to dilute solutions. Thus, for example, a solution of normal blood serum containing 1 part in 1,000 invariably gives a reliable reaction with the corresponding prepared serum.

C. A. M.

The Determination of Aloins in Aloes. E. Léger. (*Journ. Pharm. Chim.*, 1902, xv., 519-522.) For the isolation of barbaloin, isobarbaloin, and other aloins, the author boiled 500 grammes of the aloes with a mixture of 1,800 c.c. of chloroform and 600 c.c. of methyl alcohol for four hours under a reflux condenser. After settling, the supernatant liquid was decanted and distilled, and the residue taken up with absolute alcohol, from which crystals of aloins separated in three to four days. By this means from 5 to 6 per cent. of barbaloin, free from isobarbaloin, were obtained from Cape aloes, and about the same quantity from Barbadoes aloes. Curaçoa aloes yielded about 10 per cent. of aloins, consisting of approximately equal proportions of barbaloin and isobarbaloin. From Jafferabad aloes more than 20 per cent. of aloins, consisting mainly of isobarbaloin, were isolated, whilst Socotrine aloes gave only about 4 per cent., consisting almost exclusively of barbaloin, with a minute quantity of isobarbaloin. Barbaloin was identified by the characteristics of its chlor-acetyl derivative, which melts at 146·6° C. (corr.), and forms quadratic yellow anhydrous crystals only slightly soluble in hot alcohol, but very soluble in benzene. Isobarbaloin was identified by Klunge's reaction (ANALYST, xxvii., 180), and by its bromine derivative, which is slightly soluble in cold alcohol, from which it crystallizes in yellow needles. Neither of these aloins was found in Natal aloes, which, however, yielded the two analogous compounds—nataloin, and its lower homologue, homonataloin.

C. A. M.

TOXICOLOGICAL ANALYSIS.

Estimation of Arsenic in Toxicology. G. Todeschini. (*Boll. chim. farm.*, 1902, xli., 180; through *Chem. Zeit. Rep.*, 1902, 141.)—From experiments on flesh to which known amounts of arsenic trioxide had been added, the author finds that Gautier's process for the determination of arsenic, even when the improvements recommended by its inventor in 1899 are made use of, only returns one-third of the arsenic in the form of a mirror, the other two-thirds being lost. This loss is due to the fact that the carbonaceous residue retains much of the arsenic in a form insoluble in water; while if the said residue is warmed, partial reduction of the arsenic acid to arsenious acid occurs, and the latter volatilizes. When Gautier's solution is treated with sulphuretted hydrogen all the arsenic is not precipitated, probably because destruction of the organic matter is incomplete. Selmi's process is better, as only one-half the arsenic is lost. F. H. L.

Selmi's Process for the Determination of Arsenic in Toxicology. G. Giudice. (*Gazz. chim. Ital.*, 1902, xxxii. [1], 164; through *Chem. Zeit. Rep.*, 1902, 141.)—In using this process, if the apparatus is put together with rubber corks or tubes containing antimony sulphide, considerable quantities of antimony accompany the arsenic in the cold receiver, even if the temperature of the oil-bath in which the retort and first receiver are placed does not exceed 130° C., so that the mirror may consist in large part of antimony instead of arsenic. If the material under examination contains antimony compounds, antimony will contaminate the arsenic if the temperature is allowed to reach 130° C., but will not enter the cold receiver provided the oil-bath is kept below 115° C. Mercury and tin salts do not affect the working of Selmi's process, as they are not driven over even at a temperature of 130°.

F. H. L.

ORGANIC ANALYSIS.

On Herzig and Meyer's Method of Determining Methyl. M. Busch. (*Berichte*, 1902, xxxv., 1565-1567.)—The modification of Zeisel's method devised by Herzig and Meyer affords a means of determining the methyl in combination with nitrogen, as well as of that combined with oxygen, and of distinguishing between the two in certain cases. Thus, whilst the methoxyl group is hydrolyzed by being boiled with hydriodic acid, a much higher temperature (200° to 300° C.) is as a rule required to dissociate the methyl group from the nitrogen. In some cases, however, *e.g.*, 1-phenyl-4-methylanilino-urazole, the methyl is liberated from the nitrogen by the boiling acid, and the author therefore concludes that when the test gives a negative result the absence of methoxyl can be regarded as proved, but that a positive result cannot be accepted as conclusive of methylimin without further evidence. C. A. M.

Separation of Cholesterin and Phytosterin from Mixtures of Fatty and Mineral Oils. J. Marcusson. (*Mitth. Königl. Techn. Versuchsanst.*, Berlin, 1901, xix., 259; through *Chem. Zeit. Rep.*, 1902, 154.)—The author has already described (Analyst, 1901, xxvi., 302) two processes for the above purpose. The following method is specially suitable for such mixtures as contain very viscid mineral oils. It does not yield quantitative results, but if 100 grammes of the sample are taken it enables the vegetable or animal oil to be identified in presence of a mineral oil. The oil is saponified with alcoholic potash, the solution is diluted with an equal volume of water, and the mixture is shaken out several times with petroleum spirit till the mineral oil is extracted. The alcohol is driven off from the residual liquid, which is then agitated repeatedly with ethyl ether to dissolve the cholesterin and phytosterin. On distilling away the ether and recrystallizing from alcohol the cholesterols of the fats may be recovered pure in the usual manner.

F. H. L.

The Quantitative Estimation of Colophony in Fats, Oils, Soaps, Ceresin, Paraffin, etc. Holde and Marcusson. (*Mittheil. aus den Königl. techn. Versuchsanstalt.*, 1902, 40-49.)—As the amount of resin in soaps and oils cannot be accurately estimated by either Twitchell's or Gladding's processes, the authors recommend a method which is a combination of these two processes. The results of test experiments, given in the original paper, show that very accurate results can be obtained in this way. The method is as follows:

(*a*) *In Fats and Soaps containing no Unsaponifiable Matter.*—About 5 grammes of the sample (correspondingly more in the case of soap containing water) are weighed out and boiled with 50 c.c. of alcoholic potash for half an hour under a reflux condenser. The alcoholic solution is then evaporated, the residue dissolved in water and decomposed with hydrochloric acid. Soaps containing no free fat may be directly dissolved in water and decomposed with acid. The separated fatty acids are removed by shaking with ether; the aqueous acid solution is neutralized, evaporated to about 25 c.c., re-acidified and shaken out with ether. After distilling off the ether from the united ethereal extracts, the residue of fatty acids is dissolved in 50 c.c. of absolute alcohol, and the acids converted into esters by passing a moderately rapid current of dry hydrochloric acid gas through the solution cooled by ice-water to a temperature not above 10° C. When the operation is complete (it usually takes one to two hours) the flask and its contents are allowed to stand for half an hour at the ordinary temperature. The contents are then washed into a large Erlenmeyer flask with five times their volume of water, and boiled under a reflux condenser for half an hour. The cooled solution is now shaken out with 100 c.c. of ether in a separating funnel, and then with several successive quantities of 50 c.c. of ether until the extracts are colourless. The aqueous liquid is neutralized, evaporated to 50 c.c., acidified, and repeatedly extracted with small quantities (25 c.c.) of ether. In this way the water-soluble constituents of the colophony are obtained. After mixing the ethereal solutions they are shaken out with about 50 c.c. of potash solution (10 grammes of caustic potash, 10 grammes of alcohol, and 100 c.c. of water). A brown layer separates out between the ether and the alkaline solution, and is drawn off with the latter. It contains a considerable portion of the resin-soap, which is only slightly soluble in the potash solution. The ether is then well washed with water to remove soluble resin-soaps, again with two successive quantities (10 c.c.) of the potash solution, and finally with water until the washings are colourless. In the presence of fish-oil acids or much colophony the washing with water must be very thorough. To remove any fatty esters which may have been mechanically carried into the alkaline aqueous liquors, the latter are shaken with 50 c.c. of ether.

After separating the ether it is extracted with 5 c.c. of the potash solution, and the aqueous alkaline extract added to the main bulk of the same. This is now acidified and shaken out with successive quantities (50 c.c.) of ether until completely extracted. The acid solution is neutralized, evaporated to a small bulk, re-acidified, and again shaken out with ether. The total ether extracts are washed with 20 c.c. of water, and the ether is distilled off. The residue of resin-acids so obtained—still contaminated with unchanged fatty acids—is placed in a tared glass basin and evaporated with several small successive additions of absolute alcohol to remove the last traces of water, and weighed. The fatty acids still remaining in the resin-acids are removed by the employment of Gladding's process. To this end from 0·4 to 0·6 gramme of the above obtained resin-acids are placed in a 100 c.c. stoppered and graduated cylinder, and dissolved in 20 c.c. of 95 per cent. alcohol. If only a small amount of resin-acid is obtained, the following proportions of the ether-alcohol mixture must be correspondingly altered, whilst, in the case of a large yield of acids, it is advisable to dissolve the whole in so much 95 per cent. alcohol that 20 c.c. of the solution shall contain 0·5 gramme of resin-acid. A drop or two of phenolphthalein solution are added to the alcoholic solution in the cylinder, and then concentrated caustic soda solution (1 of NaOH to 2 of water), until the reaction is just alkaline. The loosely stoppered cylinder and its contents are heated for a short time in the water-bath, then cooled, ether is added up to the 100 c.c. mark, and mixed. One gramme of dry powdered silver nitrate is now added, and the contents of the cylinder are shaken for fifteen to twenty minutes to convert the resin-acids into silver salts. When the insoluble silver salts have completely separated and settled (after standing overnight if necessary), 70 c.c. of the solution are pipetted into a second 100 c.c. cylinder and shaken with 20 c.c. of dilute hydrochloric acid (1 : 2). The ether layer is drawn off, and the aqueous portion twice shaken out with 20 c.c. of ether. The united ether extracts are washed with 20 c.c. of water, filtered into a small flask, and the ether is distilled off. The residue, amounting to about 10 c.c., is transferred to a weighed dish, evaporated, dried for a short time at 110° to 115° C., and weighed. The weight of the resin-acids so found is calculated back into the first weight (impure acids) obtained, and then on the original substance taken. The percentage found is corrected by the subtraction of 0·4 per cent., this allowance being made for a small amount of fatty acid which is always present. As colophony contains an average of 8 per cent. of unsaponifiable matter, a second correction is therefore necessary, the true percentage of colophony in the substance under examination being found by the following equation, in which the two corrections are combined:

$$100 \frac{(\text{percentage of resin acids found} - 0{\cdot}4)}{92} = \text{percentage of colophony.}$$

Should more than 20 per cent. of colophony be present, it is better to directly estimate the unsaponifiable matter. The latter is contained in the ethereal solution of the fatty esters after the resin acids have been removed. After saponifying the esters by the addition of 25 c.c. of N. alcoholic potash, the solution is diluted with 150 c.c. of water and shaken out twice with 150 c.c. of ether. The main bulk of the ether is distilled off, and the remainder allowed to be evaporated at the ordinary temperature. (By warming, volatile substances are lost.) An oily residue remains, which is freed from traces of soap by treating it with a little alcoholic potash, slowly evaporating off the alcohol, and extracting this residue with petroleum spirit.

(*b*) *In Fats and Soaps containing Unsaponifiable Matter.*—A sufficient quantity of the sample is weighed out to yield about 5 grammes of fatty acids; benzene (free from thiophene) is added, and the sample is saponified. The unsaponified matter is separated from the soap solution in the usual way, and the resin and fatty acids in the latter estimated by the process given above. An accurate estimation of the unsaponifiable substances in this case is not possible, but they may be approximately calculated by assuming that the colophony found to be present itself contains on the average 8 per cent. of these substances.

(*c*) *Estimation of Colophony in Ceresin and Paraffin.*—The resin is removed from the sample by thorough extraction with boiling 70 per cent. alcohol. The united alcoholic extracts are filtered when quite cold, and the alcohol is distilled off from the clear filtrate. The residue is dried at 110° to 115° C., and weighed. Should fatty acids also be present, the residue, obtained after the distillation of the alcohol, must be treated according to the method described under (*a*). W. P. S.

Larch Turpentine and Venice Turpentine. **L. E. Andés.** (*Chem. Rev. Fett- u. Harz- Ind.*, 1902, ix., 126-128.)—*Larch Turpentine*, obtained from the trunk of the larch-tree, was formerly known also as Venice turpentine from the fact that Venice was the chief emporium for it; but this is no longer the case, and the name "Venice turpentine" is now applied to artificial mixtures of rosin, rosin oil, and turpentine used in the manufacture of sealing-wax and varnish. Larch turpentine has a pleasant odour, a bitter taste, and a yellow or brown colour. It is readily soluble in alcohol, ether, acetic acid, and acetone, and partially soluble in carbon bisulphide. It consists of a dextro-rotatory rosin and a lævo-rotatory volatile oil. According to Dieterich, it gives the following values: Acid value, 66·92 to 68·85; ether value, 46·27 to 54·94; saponification value, 114·5 to 127·71; acetyl acid value, 69·87 to 72·19; and acetyl saponification value, 178·95 to 190·86. The following results were obtained by Beckurts and Bruche in the examination of seven samples: Specific gravity, 1·060 to 1·190; acid value, 76 to 101; ether value, 0 to 9; and saponification value, 81 to 101.

Venice Turpentine.—In addition to the artificial product, there is also a commercial French product, "*térébenthine de Venise*," which is a superior variety of balsam obtained from shore pines. An artificial product examined by Dieterich had the following characteristics: Acid value, 98·79; ether value, 0·88; and saponification value, 97·66. In order to distinguish between larch turpentine and an artificial mixture, the conclusions drawn from the odour, inflammability, solubility in alcohol (90 per cent.), and analytical values may be confirmed by Hirschsohn's ammonia test. Ordinary turpentine, on treatment with five times its volume of ammonia solution (specific gravity, 0·96), gradually yields a milky emulsion, whilst in the case of larch turpentine the liquid remains clear. When the lower layer of larch turpentine is stirred it is gradually transformed into a semi-solid, opaque substance, whilst the supernatant liquid becomes slightly turbid. Ordinary turpentine, however, is immediately distributed, and the milky emulsion rapidly solidifies to a gelatinous mass. In the case of a mixture of the two in equal parts, the substance is distributed throughout the ammonia, and a solid mass, which clears on heating, is obtained in about five minutes. When the mixture only contains about 20 per cent. of ordinary turpentine the milky emulsion becomes clear on heating the tube in boiling water, but no solidification occurs. Comparative tests with genuine larch turpentine may serve to detect smaller quantities. When 1 part of the sample is shaken with 30 parts of 80 per cent. alcohol a clear and permanent solution is obtained with larch turpentine, whilst in the case of ordinary turpentine about half of the substance soon separates out. C. A. M.

The Use of Iodine Monochloride in the Determination of the Iodine Absorption of Oils. J. J. A. Wijs. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1902, v., 497-504.)—As regards the keeping qualities of the solution of iodine monochloride in glacial acetic acid, it is stated that the solution decreases in strength only to a very slight extent when carefully prepared. The presence of iodine trichloride causes a loss of strength, as does also the use of acetic acid containing water and reducing substances. In the same way ordinary Hübl's solution was found to diminish in strength according to the quality of the alcohol used in its preparation. The iodine values obtained by the employment of iodine monochloride agree with the theoretical numbers in the case of unsaturated fatty acids having only one double linking. In using Hübl's solution various precautions must be taken in order that the results may agree with those obtained by the iodine monochloride process (see ANALYST, xxiv., p. 259). W. P. S.

The Alkalinity of Crude Sugar. Lauterbach. (*D. Zuckerind.*, 1902, xxvii., 653 and 780; through *Chem. Zeit. Rep.*, 1902, 154.)—When crude sugar is stored for any length of time, bicarbonates are formed in it, while the water it contains becomes saturated with carbon dioxide. It is therefore necessary before titrating a sample for alkalinity with phenolphthalein to boil the liquid thoroughly. If this is done the results are perfectly accurate, and Herberger's objections (this volume, p. 196) to the use of that indicator are without point. F. H. L.

Identification of Sugars. E. Votocek. (*Chem. Listy*, 1902, xxvi., 122; through *Chem. Zeit. Rep.*, 1902, 141.)—The author finds that it is as easy to test for pentoses in any suitable hydrazone by distillation with 12 per cent. hydrochloric acid as in any other compound, the test being rendered additionally delicate by collecting a distillate of 180 c.c., and then redistilling it with sodium chloride, a method which enables, for example, 0·06 gramme of the methylphenylhydrazone of methylpentose to be recognised. Tested with phloroglucinol, 0·33 gramme of the benzylphenylhydrazone of arabinose gives a large greenish-black precipitate, 0·2 gramme of the corresponding compound of xylose behaving identically. 0·3 gramme of the same hydrazone of galactose yields no precipitate with phloroglucinol, while those of rhamnose and rhodeose from jalapin or convolvulin give cinnabar-coloured precipitates. In presence of furol, methylfurol may be discovered by means of resorcinol, as the gray furol resorcide does not mask the carmine colour of methylfurol resorcide. F. H. L.

A New Method for the Determination of Cellulose. S. Zeisel and M. J. Strittar. (*Berichte*, 1902, xxxv., 1252-1255.)—The authors' method is based upon the fact that cellulose in the narrower sense (dextro-cellulose) is not converted to any large extent into products soluble in dilute ammonium hydroxide, when oxidized by potassium permanganate in the presence of nitric acid, whereas the non-cellulosic constituents of wood readily yield oxidation products, part of which are soluble in water, whilst part can be extracted with 2·5 per cent. ammonium solution.

From 1 to 1·5 gramme of the finely divided material is stirred with dilute nitric acid, and a 3 per cent. solution of potassium permanganate introduced, 1 c.c. at a time, until the red colour of the liquid is plainly perceptible after thirty minutes, the oxidation taking about two hours in all. The excess of permanganate and the precipitated manganese dioxide are then destroyed by the addition of sulphur dioxide or sodium bisulphite, and the liquid filtered. The residue is washed with water, then treated for forty-five minutes with a 2·5 per cent. solution of ammonia at 60° C., and again washed successively with hot water, alcohol, and ether.

In this way oak raspings were found to contain 37·2 per cent. of cellulose. The results were concordant, but were much lower than those obtained by Schulze's chlorate process, although showing a satisfactory agreement with those given by the tedious process of Schulze and Henneberg, in which the wood is extracted with water and alcohol, and oxidized for days with a solution of potassium chlorate in nitric acid.

About 30 per cent. of the cellulose is converted into oxycellulose by oxidation with permanganate, but the slight error thus introduced can be corrected by extracting the residual cellulose with a boiling 10 per cent. solution of sodium hydroxide, in which the oxycellulose dissolves. The amount of soluble compounds formed from the cellulose in the oxidation does not exceed 4 per cent. Hemi-celluloses are completely converted into soluble oxidation products. C. A. M.

Detection of Pentoses in Urine in the Presence of Glycuronic Acids. K. von Alfthan. (*Arch. Experiment. Pathol.*, 1902, xlvii., 417; through *Chem. Zeit. Rep.*, 1902, 154.)—On treatment with benzoyl chloride and sodium hydroxide both pentoses and glycuronic acids are converted into benzoyl esters. When these are saponified with sodium ethoxide the glycuronic acids separate out, the pentoses remaining in solution. If, then, the filtrate yields the phloroglucinol or orcinol test, pentoses must be present in the urine. F. H. L.

A Reagent for Albumin in Urine. Pollacci. (*Giorn. di farm. di Trieste*, 1901, 235; *Ann. de Chim. anal.*, 1902, vii., 195.)—The reagent consists of 1 gramme of tartaric acid, 5 grammes of powdered mercuric chloride, and 10 grammes of pure sodium chloride dissolved in 100 c.c. of water, and mixed with 5 c.c. of formalin.

Two c.c. of this reagent are placed in a test-tube, and 3 to 4 c.c. of the urine under examination carefully poured down the side of the tube so as to avoid mixing the liquids. In the case of pathological urine, a white zone is immediately produced at the junction of the liquids, whilst normal urine does not give any reaction until after a lapse of ten to fifteen minutes.

The limit of sensitiveness of the reaction is 1 in 370,000. C. A. M.

Valuation of Rubber Goods. O. Mayer. (*Chem. Zeit.*, 1902, xxvi., 481.)—In this article the author points out that there is no method which can be considered universally applicable to the examination of indiarubber goods. Heintz has not submitted sufficient evidence to prove that the substance left after the various foreign ingredients have been removed by appropriate solvents agrees with the formula $(C_{10}H_{16})_n$; indeed, in view of the way in which caoutchouc may be decomposed during its incorporation with "substitutes," etc., and of its liability to change during the extraction of those substitutes, such a method of ultimate analysis as is recommended by Heintz (this volume, p. 200) is not likely to yield trustworthy results. Even the Henriques process cannot be regarded as a routine method, for it needs modification to suit different grades of material; and there are, in fact, on the market now certain descriptions of rubber wares which are not amenable to the Henriques treatment without considerable alteration in the usual procedure. Mayer considers it unfortunate that manufacturers have been led to believe that reliable methods of valuation have already been elaborated. F. H. L.

A Reaction distinguishing between α- and β-Naphthols. A. Jorissen. (*Ann. de Chim. anal.*, 1902, vii., 217-219.)—A pinch of the naphthol is treated with about 2 c.c. of an aqueous solution of iodine and potassium iodide, and the liquid shaken with an excess of an aqueous solution of sodium hydroxide. A clear, colourless liquid is obtained with β-naphthol, whilst in the case of α-naphthol there is turbidity and an intense violet coloration. When mixtures of the two naphthols are thus tested the liquid assumes a more or less pronounced violet colour. C. A. M.

The Volumetric Determination of Thymol. E. Zdarek. (*Zeit. anal. Chem.*, 1902, xli., 227-331.)—This is an application of Koppeschaar's method of determining phenol, the thymol being precipitated by bromine, and the excess of the latter titrated with sodium thiosulphate after the addition of potassium iodide and starch solution. The solutions employed by the author are the same as those of Koppeschaar, with the exception of the bromine solution, which is of twice the strength. They are as follows: (1) An aqueous solution of sodium thiosulphate (9·76 grammes per litre), corresponding to a solution of iodine containing 5 grammes per litre; (2) an aqueous solution of potassium iodide containing 125 grammes per litre; (3) an aqueous solution containing in a litre 3·571 grammes of dry sodium bromate and 12·178 grammes of dry sodium bromide. On mixing 25 c.c. of this solution with 10 c.c. of the iodide solution (2) and 5 c.c. of concentrated hydrochloric acid, the liberated iodine is equivalent to 90 c.c. of the sodium thiosulphate solution (1).

In making a determination, the dry thymol is weighed into a flask provided with a stopper, and for each 0·1 gramme 20 c.c. of the bromide solution (3), and 4 c.c. of concentrated hydrochloric acid are added. The flask is well shaken for about five minutes, after which 10 c.c. of the potassium iodide solution and a little starch solution or paste are introduced, and the contents immediately titrated with the thiosulphate. According to the results of the author's analyses, 1 molecule of bromothymol contains 4 atoms of bromine. In ten test determinations thus made, the amounts of thymol found varied from 98·6 to 100·2 per cent. of the theoretical quantity. The bromine compound gradually loses bromine when kept in a desiccator over sulphuric acid, until finally a stable compound corresponding to dibromothymol is left. On treating thymol with a large excess of bromine an oily compound was formed, which, when kept for some days in a sealed glass tube, yielded a deposit of nearly colourless crystalline needles, melting at 71° C., and having the composition of a thymol containing 5 atoms of bromine.

The author has used this method to determine the solubility of thymol, and has found that 1 gramme dissolves in 1176·4 c.c. of water at 19·4° C. At 15° to 20° C. 1 part of thymol is soluble in 0·24 to 0·28 part by weight of strong alcohol (90 to 91·2 per cent. by volume), in 0·22 to 0·26 part of ether, and in 0·67 to 0·7 part of chloroform.

C. A. M.

Colour Reaction for Thiophen. H. Kreis. (*Chem. Zeit.*, 1902, xxvi., 523.)—If a very weak solution of thalline base (tetrahydro-*p*-oxyquinoline methyl ester) in petroleum spirit is shaken with a liquid containing thiophen and with some 1·4 nitric acid, a distinct, but transient, violet colour is produced, which gradually becomes reddish, and finally yellow. Addition of water destroys the colour, so that weaker nitric acid must not be employed; stronger acid is also to be avoided. The test can be used to show either thiophen or thalline in very minute quantities. It is very convenient for the detection of thiophen in benzene, and it serves equally for the recognition of methylthiophen in toluene. Other alkaloids shaken with benzene containing thiophen and nitric acid do not yield noteworthy colour reactions. Bellier's test for sesamé oil (ANALYST, 1900, xxv., 50), which is somewhat similar, does not, however, depend on the presence of thiophen.

F. H. L.

The Volumetric Determination of Disodium Methyl-arsenate. E. Falières. (*Journ. Pharm. Chim.*, 1902, xv., 466-469.)—Silver methyl-arsenate is sufficiently soluble in water to prevent an exact gravimetric determination of the sodium salt by precipitation with silver nitrate. It is, however, completely insoluble in a $\frac{N}{10}$ or $\frac{N}{50}$ solution of silver nitrate, and on this fact the author bases his method of estimation.

Disodium methyl-arsenate crystallizes with 6 molecules of water, which it loses at 120° to 130° C., but regains to a large extent on exposure to the air. The author prefers to take the formula, $As(CH_3)O_4Na_2.6H_2O$, as typical of the salt, since this does not alter in composition after an exposure of several days to the air.

In the volumetric method 0·2 gramme of the crystalline salt (from which chlorides, sulphates, arsenites, arsenates, phosphates, carbonates, and iodides have been proved to be absent) is dissolved in 10 c.c. of water, and the solution mixed with 40 c.c. of $\frac{N}{10}$ silver nitrate solution, and rapidly filtered. The filtrate is used to titrate 10 c.c. of $\frac{N}{10}$ sodium chloride solution diluted with 30 c.c. of water, and the amount of silver nitrate still present is thus found.

The amount of disodium methyl-arsenate corresponding with the amount of silver nitrate used in the precipitation is then found by means of the formula

$$\frac{292 \times N \times 5 \times 100}{340},$$

where N represents the weight of silver nitrate consumed, 292 the molecular weight of disodium methyl-arsenate, and 340 twice the molecular weight of silver nitrate.

C. A. M.

The Precipitation of Alkaloids, Metallic Salts, and Proteids by Extracts of Coffee and Tea. T. Sollmann. (*Journ. Med. Research*, Ohio, 1902, 43-53; through *Amer. Journ. Pharm.*, 1902, lxxiv., 299-303.)—According to the results of the author's experiments, the reactions given by tea tannin closely resemble those of gallotannic and quercotannic acid, and differ greatly from those given by coffee tannin (a diglycosyl ester of cinnamic acid). The extracts used in the experiments were prepared by boiling the tea or roasted coffee for forty-five minutes with 10 parts of water, and filtering the liquid. Neither extract gave any precipitate with Mayer's reagent or with hydrochloric acid in the dilution in which they were used in the tests. The proportions usually employed in the case of alkaloids were 1 c.c. of the decoction to 2 c.c. of a 1 per cent. aqueous solution of the alkaloid or its salt, or 5 c.c. each of the alkaloidal solution (1 : 1,000), and of the decoction.

Alkaloids.—Atropine, coniine, morphine, and pyridine were not precipitated, even from a moderately concentrated solution by coffee, but were precipitated by tea from strong solutions. Aconitine, brucine, cocaine, lobeline, nicotine, and pilocarpine were sparingly precipitated from weak solutions by tea, but not by coffee, even when they were present in strong solutions. Apomorphine, the cinchona alkaloids, hydrastine, strychnine, and veratrine were precipitated by both tea and coffee from weak solutions. None of the alkaloids except apomorphine was completely precipitated.

Metallic Salts.—Tea was found to effect a more complete precipitation than coffee. Aluminium, lead, and silver were precipitated practically completely by both, whilst mercury was partially precipitated by tea, but not by coffee.

Proteids (*Egg-white, Albumose, Gelatin*).—A sharp differentiation between the tannins of tea and coffee could be effected by means of these proteids. The tea decoction gave a large precipitate with them, whereas the coffee decoction was unaffected, or, at most, rendered slightly turbid.

C. A. M.

The Kjeldahl Process. **K. Neuberg.** (*Beitr. chem. Physiol. u. Pathol.*, 1902, ii., 214; through *Chem. Zeit. Rep.*, 1902, 141.)—In order to destroy the amido-mercuric sulphate which is produced in the Kjeldahl flask when mercury is employed, the author recommends sodium thiosulphate. This has the advantage of being fit for use in the solid form, so that dilution of the liquid is avoided. F. H. L.

INORGANIC ANALYSIS.

A Note on the Volumetric Determination of Copper. **S. W. Parr.** (*Journ. Amer. Chem. Soc.*, xxiv., 580.)—In the method depending on the oxidation by permanganate of cuprous thiocyanate (*Journ. Amer. Chem. Soc.*, xxii., 685) the end-reaction is sometimes unsatisfactory. This can be obviated by the following modification: The washed precipitate of cuprous thiocyanate and the asbestos pulp are returned to the original beaker, 10 c.c. of a 10 per cent. potassium hydroxide solution and 10 c.c. of ammonia (specific gravity, 0·96) are added, and potassium permanganate solution is immediately added until, upon warming to 50°, a green colour remains. About one-third or one-fourth of the quantity of permanganate necessary for this is then run in, and the whole allowed to stand for five minutes, acidified with 25 c.c. of sulphuric acid (1 : 1 or 2), and the titration finished as usual. The results obtained with this method are very good. To calculate results, the iron value of the permanganate is multiplied by the factor 0·1602. A. G. L.

The Volumetric Determination of Copper by means of Potassium Iodide. **F. M. Litterscheid.** (*Zeit. anal. Chem.*, 1902, xli., 219-227.)—On treating a solution of a copper salt with sulphur dioxide, and then adding potassium iodide in slight excess, the copper is precipitated quantitatively as represented in the equation

$$2CuSO_4 + 2KI + SO_2 + 2H_2O = Cu_2I_2 + 2KHSO_4 + H_2SO_4.$$

The solution of the copper salt, which should be neutral or only slight acid, and should contain not less than 0·1 gramme of copper, is mixed with a few c.c. of sulphur dioxide solution, and then with a slight excess of decinormal potassium iodide solution. After standing for an hour for the cuprous iodide to become crystalline, the liquid is made up to definite volume and filtered, and an aliquot portion of the filtrate mixed with an excess of decinormal silver nitrate solution, and then with 10 c.c. of dilute nitric acid, and titrated with decinormal thiocyanate solution, with iron ammonium sulphate as indicator. Lead, mercury, and other metals precipitated under these conditions must be absent, and also substances that liberate iodine from potassium iodide.

Copper and Silver.—About 1 gramme of the alloy is dissolved in nitric acid, and the solution mixed with about 5 c.c. of dilute sulphuric acid, and evaporated nearly to dryness. It is then diluted to about 50 c.c., and any separated silver sulphate brought into solution by warming. When cold, 1 drop of a 5 per cent. solution of potassium nitrite and some starch paste are introduced, and the silver titrated with decinormal potassium iodide. The liquid is then filtered, and the copper determined in the filtrate as described above.

Copper and Bismuth.—The solution of the alloy is concentrated to a small volume, the bismuth separated as basic carbonate by the addition of ammonium carbonate and ammonia, and the copper determined in the filtrate. The method is also applicable to the determination of nickel and zinc in the presence of copper. When lead is present, it is first determined in the form of sulphate. C. A. M.

Preparation of Arsenic-Free Zinc. Otto Hehner. (*Jour. Soc. Chem. Ind.*, 1902, xxi., 675.)—Zinc suitable for use in the Marsh-Berzelius test is prepared as follows: Melt about a pound or two of ordinary block-zinc, no matter how impure, in a clay crucible over a good gas fire. When quite fluid, throw into the metal a piece of sodium, taking for each pound of zinc about 1 gramme of sodium at a time. Stir the molten metal with a piece of hard-glass tubing bent at right angles. A black scum forms immediately, which is removed with a china spoon or a crucible lid held in the tongs. Stir vigorously, and remove the scum from time to time till the sodium appears to have oxidized out; then add another piece of sodium, stir and skim as before, and oxidize well. This takes altogether about ten minutes. Finally, pour out the metal into a second clean clay crucible, and repeat the sodium treatment once more. It is best to keep two crucibles with file-cuts for the two stages. Allow the molten metal to cool considerably before granulating, as when the zinc is very hot and fluid heavy drops like shot are obtained, whereas when the zinc is near its solidifying point, thin flakes, presenting a large surface to the acid, are produced. A. M.

The Electrolytic Determination of Bismuth. O. Brunck. (*Berichte*, 1902, xxxv., 1871-1873.)—Winkler's method (*Berichte*, xxxii., 2192), in which wire gauze is employed as the cathode, has been found by the author to yield good results in the determination of bismuth. The metal or its salt is dissolved in nitric acid, the amount of which may be from twenty to twenty-five times that of the bismuth, though its strength must not exceed about 2 per cent. Another essential condition is that the tension of the current must not exceed 2 volts. The permissible strength of the current varies with the concentration of the solution. Thus, if more than 0·1 gramme of bismuth in 100 c.c. be present, a current of 0·5 ampère or more may be employed, whilst with less than 0·05 per 100 c.c., a current not exceeding 0·1 ampère is best. The electrolyte is first heated to 70°–80° C., so that its resistance is lessened, and it is possible to transmit a fairly large current, notwithstanding the low tension. As the solution cools and becomes poorer in metallic ions the strength of the current diminishes, until at length it falls to only a few hundredths of an ampère. The deposition of even large quantities of bismuth is complete in two to three hours, and when the above conditions are observed the gray deposit is compact and adherent. After completion of the electrolysis both electrodes are transferred to a beaker of water, and the current continued for a short time, in order to effect the deposition of any bismuth dissolved by the adherent nitric acid. Finally, the cathode is washed successively with water, alcohol, and ether, and weighed. The results of the test experiments described agree well with the theoretical amounts.

Sulphuric acid alone is not a suitable solvent for the metal, owing to its being necessary to use such large quantities to prevent the formation of basic salts. All the attempts to separate lead from bismuth by this means were unsuccessful, the lead peroxide invariably containing more or less bismuth. C. A. M.

On the Compounds of Tellurium with Bismuth and the Quantitative Separation of the Two Elements. A. Gutbier. (*Zeits. Anorg. Chem.*, xxxi., 331.)—In the analysis of various mixtures of bismuth and tellurium prepared by himself, the author has examined three methods of separating these two elements. He condemns the first, in which tellurium is precipitated by sulphurous acid, bismuth being thrown down by hydrogen sulphide in the filtrate; and the second, in which bismuth is first precipitated as oxychloride, as being inexact. He obtained good results, however, with the third method, which depends on the solubility of tellurium sulphide in alkaline sulphides. The method was carried out as follows: The powdered substance was dissolved in aqua regia, and the solution repeatedly evaporated on a water-bath with hydrochloric acid until the smell of chlorine had disappeared. After diluting with water as far as possible without causing bismuth oxychloride to separate, and heating to 60° to 70°, the solution was saturated with hydrogen sulphide, the precipitate allowed to settle, and then filtered through a weighed filter-paper placed in a funnel with ground edges, and having a stopcock fitted to its stem. The filtrate and washings were concentrated and again treated with hydrogen sulphide, but one precipitation was generally found to be sufficient to throw down the whole of the elements.

The precipitated sulphides were then treated on the filter with warm potassium sulphide solution until all the tellurium was extracted, the residue washed thoroughly with water, finally with alcohol and carbon bisulphide, dried at 100°, and weighed as Bi_2S_3. The tellurium was precipitated in the filtrate by cautiously adding hydrochloric acid and gently heating. After filtering off, the precipitate was dissolved in fuming nitric acid, the solution evaporated repeatedly on the water-bath with hydrochloric acid, the residue dissolved in water and a little hydrochloric acid, and boiled with an excess of barium bicarbonate solution to remove sulphuric acid. In the filtrate from the barium sulphate and carbonate, tellurium was precipitated by any one of the usual methods and weighed as Te. The author finds it best to use a Neubauer's (platinum) crucible for the filtration and drying of the tellurium, on account of the ease with which it oxidizes. A. G. L.

A New Qualitative Test for Cobalt. J. L. Danziger. (*Journ. Amer. Chem. Soc.*, xxiv., 578.)—About 5 c.c. of the solution to be tested is acidified with hydrochloric acid, solid ammonium thioacetate added, followed by a few drops of stannous chloride, and an equal volume of amyl alcohol; mixtures of acetone and ether, or alcohol and ether, may also be used, though less efficiently. The whole is well shaken and allowed to stand. If cobalt is present the upper layer will be coloured an intense blue from the formation of the double salt $(CH_3COS)_2Co, 2CH_3COSNH_4$. The test is as sensitive as Vogel's (ANALYST, xxvi., 218), and will serve to detect 1 part of cobalt in 500,000 parts of water. The object of adding stannous chloride is to reduce any ferric iron, which would give a deep red colour with the reagent. A. G. L.

Estimation of Titanium in Steel and Iron. G. B. Waterhouse. (*Chem. News*, lxxxv., 198.)—For the estimation of titanium in steels or pig-irons 5 grammes of the drillings are dissolved in 50 c.c. of strong hydrochloric acid, the solution is evaporated to dryness, the residue baked, heated for a few minutes with about 20 c.c. hydrochloric acid, and the solution boiled, after the addition of 70 c.c. water, until all the chlorides are dissolved. The solution is then filtered, the insoluble matter thoroughly washed with dilute hydrochloric acid, and the volume of the filtrate made up to about 150 c.c. Dilute ammonia is then added till a small, permanent precipitate is obtained, which is dissolved with a few drops of hydrochloric acid, after which 50 c.c. of a 20 per cent. solution of sodium sulphite are slowly added, any precipitate or dark colour being removed with a little dilute sulphuric acid. The solution is then heated to boiling, a hot solution of 50 c.c. glacial acetic acid and 20 grammes sodium acetate added, and the whole boiled briskly for fifteen minutes. The precipitate formed is filtered off, thoroughly washed, and ignited together with the first insoluble residue.

The oxides obtained are fused for several minutes with 10 grammes of sodium carbonate; after cooling the melt is treated with 150 c.c. hot water, the precipitate well washed, transferred to a beaker, any adhering precipitate being dissolved in a little hydrochloric acid, and, after adding 10 c.c. dilute sulphuric acid, the solution is boiled down until sulphuric acid fumes come off, allowed to cool, diluted to 50 c.c., and filtered. The filtrate is made up to a volume of about 150 c.c., made slightly alkaline with ammonia, then just acid with hydrochloric acid, and, after adding 20 c.c. sodium sulphite solution, heated to boiling, when acetic acid and sodium acetate are added as before. After boiling for fifteen minutes, the precipitate is filtered off through a large double filter, well washed, ignited, and weighed as TiO_2.

The above method was tested on samples of iron to which known quantities of a standard titanium solution had been added, and gave good results, even in the presence of chromium, aluminium, nickel, tungsten, molybdenum, or vanadium, the largest error being found in the presence of 1·1 per cent. of vanadium, in which case a percentage of 1·048 Ti, instead of 1·016, was found. A. G. L.

The Electrolytic Determination of Vanadium. P. Truchot. (*Ann. de Chim. anal.*, 1902, vii., 165-167.)—This method is based upon the fact that a hot, slightly ammoniacal solution of sodium vanadate is decomposed by an electric current, with the deposition of hydroxide or a mixture of hydroxides of vanadium on the cathode.

The solution of the salt, which in the author's experiments contained from 0·012 to 0·05 gramme of V_2O_5 in 200 c.c. was rendered slightly alkaline with 20 to 30 drops of ammonium hydroxide, heated to 80° to 85° C., and electrolyzed with a current of 2 to 2·5 volts and 0·3 ampère for eight to ten hours, the liquid being meantime kept at the same level by the addition of ammoniacal warm water. The platinum cone with the deposit was then washed with water and with alcohol, and ignited in a muffle for a few minutes to convert the hydroxides into vanadic anhydride (V_2O_5), allowed to cool slowly in a dry atmosphere, and weighed.

The results thus obtained were within 0·0005 gramme of the theoretical amounts.

C. A. M.

INSTITUTE OF CHEMISTRY OF GREAT BRITAIN AND IRELAND.

The Council of the Institute of Chemistry has decided that examinations for persons desirous of qualifying themselves to be public and technical analysts shall be held in Dublin in the month of July, 1903.

The examinations will be open only to candidates who have complied with the regulations of the Institute.

The following is a list of the names of the candidates who passed the examination of the Institute of Chemistry held in July, 1902:

Intermediate Examination.—Twenty-four candidates presented themselves; the following (twelve) passed: W. L. St. J. Alton, University College, London, and under M. W. Blyth, F.I.C. M. Barrowcliff, University College, Notts. P. H. Carpenter, Finsbury Technical College. R. W. Clarke, Finsbury Technical College. O. C. M. Davis, University College, Bristol. J. F. P. Fielding, King's College, London. G. W. Glen, Glasgow and West of Scotland Technical College. B. F. Howard, Finsbury Technical College. F. T. Jewson, University College, Notts. W. H. Keys, University of Birmingham. T. H. Lloyd, University College, Cardiff. E. Smith, Glasgow and West of Scotland Technical College, and under Dr. A. Thomson, M.A., F.I.C.

Final A.I.C. Examination.—Branch "A" (*Mineral Chemistry*): Seven candidates presented themselves; the following (three) passed: A. E. Garland, Associate Royal College of Science, London. J. L. Garle, University College, London. G. C. Jones,* Associate City and Guilds Institute, London. Branch "B" (*Metallurgical Chemistry*): Two candidates presented themselves and passed: W. B. Parker, University of Birmingham. J. C. Sheldon, University of Birmingham, and under A. E. Tucker, F.I.C. Branch "C" (*Physical Chemistry*): Two candidates presented themselves; one passed: H. Bassett, B.Sc. Lond., University College, London. Branch "D" (*Organic Chemistry*): Six candidates presented themselves; one passed: S. Andrews, B.Sc. Lond., Associate Royal College of Science, London. Branch "E" (*Analysis of Food and Drugs, including Examination in Therapeutics, Pharmacology, and Microscopy*): Eight candidates presented themselves; the following (five) passed: G. J. Alderton, King's College, London. R. Glegg, B.Sc. Aberdeen. E. V. Jones, University of Birmingham. W. H. Roberts, M.Sc., Victoria University College, Liverpool. H. P. Stevens,* B.A. Oxon., Ph.D. Heidelberg.

The examiners in General Theoretical and Practical Chemistry were Dr. Bernard Dyer, F.I.C., and Dr. W. Palmer Wynne, F.R.S., F.I.C. The examiner in Therapeutics, Pharmacology, and Microscopy was Dr. Arthur P. Luff, F.R.C.P., F.I.C.

* For fellowship (F.I.C.).

1902

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VOLUME XXVII.—No. 318.
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THE ANALYST.

SEPTEMBER, 1902.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

ESTIMATION OF PLATINUM, GOLD AND SILVER IN ALLOYS. ✓

By Percy A. E. Richards, F.I.C.,

(*Read at the Meeting, June* 4, 1902.)

The need for a simple and satisfactory method for the estimation of the percentage of platinum in samples of platinum alloy has long been experienced by dental metallurgists. Various processes have from time to time been suggested, but most of these entail considerable difficulty in obtaining accurate results.

The following experiments were carried out to discover, if possible, a process that should be at the same time both speedy and accurate.

(*a*) Alloys containing Platinum and Silver only.

In the first place, a weighed quantity of the alloy was boiled with aqua regia in a small flask until action ceased, but the result obtained was far from satisfactory, as the formation of the silver chloride retarded, and finally stopped, the action before it was nearly completed.

Another piece of alloy of known weight was then boiled with strong nitric acid in a flask until action ceased, and the addition of more acid produced no effect. The contents of the flask were diluted with water and the platinum filtered off and washed. It was at once evident, however, that a certain portion of the platinum had dissolved with the silver, whilst further examination showed that some of the silver had been retained in the mass of the platinum.

An endeavour to meet this difficulty was made by fusing a weighed portion of the alloy with two and a half times its weight of silver, the button so obtained being beaten out, rolled into a cornet, and boiled with nitric acid as before. Under these conditions, it has been stated, the whole of the platinum will dissolve with the silver. This, however, does not appear to be the case, as it was found that only a small percentage of the platinum dissolved, the greater portion remaining as a black powder. This process was repeated, using larger proportions of silver in the initial fusion, viz., four and six times the weight of the original alloy, but in each case the bulk of the platinum remained insoluble, although a considerable portion had dissolved. This necessitated the separation of the soluble platinum from the silver, and to effect this the filtrate and washings were heated to boiling, a little strong hydrochloric acid added gradually until precipitation ceased. The silver chloride was filtered off, well washed with water, dried and weighed. The filtrate from the silver was evaporated just to dryness, a few drops of water added, and the platinum precipitated with ammonium chloride in the presence of alcohol. The platinum was obtained from this by heating in a crucible in the ordinary way, its weight being added to that of the major portion yielded in the first stage. The fact that the platinum is only partially soluble under these conditions militates against the success of the estimation, and there is also the possibility of loss during the gentle heating of the bulk of the metal for the purpose of drying it. The experiments showed that after the nitric acid treatment the metal was rendered sufficiently explosive to frequently eject the contents from the crucible during the drying process.

These objections sufficed to condemn the method, and strong sulphuric acid was next used in place of nitric acid.

As before, a small piece of the alloy (about 0·3 gramme) was weighed, placed in a parting flask, about 10 c.c. of strong pure sulphuric acid added, and the flask heated for about fifteen minutes over a Bunsen burner until action had apparently ceased for quite a couple of minutes. The acid was allowed to thoroughly cool, and was then poured off the platinum into a beaker containing distilled water, the metal being again treated with strong sulphuric acid (about 5 c.c. were found sufficient). The flask and contents were again heated for five or ten minutes, and the acid poured off and added to the first quantity; owing to the great density of platinum this was effected without any difficulty. The metal was washed twice with water, the washings being added to the silver sulphate solution, and next with several quantities of water to free it from acid.

The platinum was then transferred to a crucible by filling the flask completely full of water and inverting over the latter, in which there was also a little water, the metal thereby falling into the crucible without loss. The greater part of the water was decanted off, and the metal dried in the air-bath before weighing.

The results obtained by this process were very satisfactory, as shown in the following table:

Alloy taken.	Platinum found.	Platinum per Cent.
0·384 gramme	0·128 gramme	33·5
0·432 „	0·144 „	33·4
0·357 „	0·119 „	33·4
0·395 „	0·139 „	33·5

The platinum residue was tested for silver, but was found to contain a minute trace only. The platinum at the end of the experiment was not disintegrated to powder as one should expect, but retained more or less its original shape, presenting, however, a decidedly blistered appearance. The drying was effected without any of the explosive phenomena observed in the nitric acid process.

The silver may be estimated in the acid filtrate, if desired, by the well-known sulphocyanide method, or, in the case of dental alloy, taken by difference. The metal can also be recovered in the following simple way: The diluted silver sulphate solution, rendered slightly alkaline with ammonia, is warmed with a little glucose solution, and the precipitated silver filtered, washed, and dried.

(*b*) ALLOYS CONTAINING GOLD, PLATINUM, AND SILVER.

For the purposes of experiment, various alloys were prepared containing a known percentage of gold, platinum, and silver. Each ingot was filed, and weighed portions of the filings used for analysis, the weights taken varying from 0·3 gramme to 0·6 gramme.

The first process employed consisted in heating the alloy to a moderate temperature in an atmosphere of chlorine under various conditions, but the results obtained were not always accurate, as the silver chloride formed was apt to fuse and prevent complete chlorination taking place.

The alloy was next treated with boiling sulphuric acid as described previously, and this method proved much more satisfactory. After the acid extraction the residual gold and platinum was washed, dried, and weighed as a check on the figures obtained. The residue was then dissolved in nitro-hydrochloric acid, concentrated to a small bulk, and the platinum precipitated as ammonia-platinum chloride, the latter strongly heated and the metal weighed.

The gold in the filtrate from the ammonia-platinum chloride was thrown down by ferrous sulphate, washed, dried, and weighed. For the reasons given above, treatment of the original alloy with aqua regia was found to be quite unsuitable.

The following results were obtained from the analysis of one of the alloys:

	Platinum.	Gold.	Silver.
No. 1	5·65	26·62	67·84
„ 2	5·51	26·47	67·92
„ 3	5·26	26·73	67·81

(*c*) ALLOYS CONTAINING GOLD, PLATINUM, SILVER, AND TIN.

The method of analysis that gave the best results in this case was practically identical with that last described.

A weighed portion of the alloy filings was extracted with boiling sulphuric acid several times as before, silver and tin being dissolved, whilst platinum and gold remained behind. The residue, well washed and dried, was weighed, the platinum and gold being afterwards separated and estimated as before. The silver was determined by titrating the sulphuric acid solution, and the tin calculated by difference.

Analysis of an alloy examined in this way gave the following figures:

	Platinum.	Gold.	Silver.	Tin.
No. 1	2·20	9·30	58·52	29·98
„ 2	2·29	9·18	58·36	30·17

Attempts to use aqua regia to dissolve the alloy resulted in the formation of purple of Cassius with the silver chloride, and this effectually prevented a satisfactory estimation.

Heating the alloy in a tube through which a stream of chlorine was passed gave fair results, the tin chloride in this method being volatilized and estimated by difference, as attempts to collect it in water, with a view to precipitation, were not very successful.

The sulphuric acid process does away with the necessity for using chlorine, and this is a distinct advantage, whilst the results obtained are certainly better.

DISCUSSION.

Mr. ALLEN said that many years ago he had had occasion to examine alloys of a somewhat similar character to those mentioned by the author, and he had then found nitric acid did not dissolve the whole of the platinum from a platinum alloy, although the contrary had been stated to be the case. One particular dental alloy of tin and gold, on treatment with nitric acid, left a residue of a purple colour, consisting either of purple of Cassius or a mixture of SnO_2 and finely-divided gold. On subsequent treatment with strong sulphuric acid this dissolved, leaving metallic gold, and yielding a solution of stannic sulphate. Stannic sulphate was analogous to, though less stable than, titanic sulphate. It could be kept indefinitely in solution in strong sulphuric acid, but if the solution were diluted with water, it gradually decomposed, even at the ordinary temperature, while on boiling it decomposed immediately into metastannic acid and sulphuric acid. That afforded a possible means of analysis, which he had found valuable at the time.

ON THE ACTION OF BOILING HYDROCHLORIC ACID UPON ARSENIC ACID.

By OTTO HEHNER.

(*Read at the Meeting, June* 4, 1902.)

IN 1862 R. Fresenius published under the above title, in the first volume of the *Zeitschrift fuer Analytische Chemie*, p. 447, a paper in which he showed that, on boiling sodium arsenate, "free from every trace of arsenious acid," with strong hydrochloric acid, small but distinct quantities of arsenic were volatilized, the distillates giving, with hydrogen sulphide, slight yellow precipitates of the sulphide. With hydrochloric acid diluted with twice its bulk of water no arsenic could be detected by hydrogen sulphide in the first portions of the distillate, while the last portions gave evidences of volatilized arsenic.

This statement is referred to in Watts' Dictionary, first supplement, p. 222, published in 1879, and has generally gone into the literature on the subject. Thus Fresenius himself, in his "Quantitative Analysis," fifth edition, warns against evaporating arsen*ic* solutions with hydrochloric acid unless care be taken often to replace the evaporating water to keep the acid from concentrating beyond a certain point. Otto, in his "Ausmittelung der Gifte," 1875, states that it is well known that on distilling strong hydrochloric acid containing arsen*ic* acid much arsenic is volatilized as chloride. As recently as this year the statement is made (Erdmann, "Inorganic

Chemistry" [German], p. 368), "Concentrated hydrochloric acid partly reduces arsenic acid on heating"; and on p. 373, "Arsenic acid is but incompletely converted into arsenic trichloride" (on treating with NaCl and H_2SO_4).

In view of these statements it seemed improbable, to say the least, that hydrochloric acid could be readily obtained free from arsenic by adding an oxidizing agent, like permanganate or bichromate, and distilling, as stated in the recent discussion on this subject.

To satisfy myself I prepared some arsenic acid, perfectly free from arsenious acid, by evaporating 3 grammes of arsenic trioxide four times with strong nitric acid. The product, freed by evaporation on the water-bath from all but traces of nitric acid, was dissolved in 100 c.c. of water; 10 c.c., corresponding to 0·3 gramme of arsenic oxide, were distilled with 100 c.c. of pure, strong hydrochloric acid. The distillate was collected in fractions, and these were tested with hydrogen sulphide; all gave strong yellow colorations, a precipitate of arsenic sulphide gradually separating. A similar result was obtained when potassium permanganate had been added before the beginning of the distillation—in other words, when the distillation was commenced in the presence of free chlorine.

This result appeared fully to corroborate the previous knowledge of the subject, and it seemed probable that hydrochloric acid might be able to reduce other mineral substances, for it is a long-known fact that some organic substances, like quinones, are reducible by hydrochloric acid.

On boiling a solution of ferric chloride (free from ferrous salt) with hydrochloric acid, both without and with the addition of permanganate, the residue in the distilling-flask showed a strong reaction for ferrous iron by means of potassium ferricyanide.

In order to prevent, in the above experiment, any mechanical carrying over of arsenic from the distillation-flask, I had fitted to it a long fractionation-tube by means of a cork. As, however, it seemed possible that the organic matter of the cork might have played a part in the reducing actions observed, I repeated the experiments with a similar apparatus having nothing but ground-glass connections. Under these conditions no formations of ferrous salts could be observed from ferric chloride, ferricyanide giving no trace of a blue coloration. In the case of arsenic acid the distillates showed no trace of any discoloration with hydrogen sulphide. This was the test solely relied upon by Fresenius, upon whose authority the statements in the books are founded. He used in his experiments a tubulated retort, with its neck turned upwards, but he does not say whether the tubulus or the retort was closed with a cork or a glass stopper. In 1862 stoppered retorts were not so usual as they are now, and, from the internal evidence, I would say that a cork closure had been employed. It seems clear from his paper that he had no suspicion that organic matter might play an important part in the reaction, for he states that, when organic substances have been "destroyed" by hydrochloric acid and chlorate, no fear of volatilization of arsenic need be entertained, provided that the said acid be kept sufficiently diluted.

The whole of the distillates, which failed to show arsenic by hydrogen sulphide, gave, however, plain indications of the presence of traces of arsenic by the Marsh-

Berzelius method. No fraction could be obtained that was satisfactorily free from arsenic. Nevertheless, the remarkable diminution in the amount of arsenic volatilized when the flask was connected with the fractionation-tube by glass only, as compared with cork connection, shows clearly that the reduction has been mainly, if not entirely, brought about by the presence of organic matter. The traces of volatilized arsenic observed even when the most scrupulous care had been taken to exclude organic matter, all parts of the apparatus having been thoroughly treated with strong sulphuric acid and potassium bichromate, are almost certainly due to the reducing action of organic dust. We have to deal in the case of the Marsh-Berzelius indication with small fractions of hundredths of milligrammes, and the dust in the air of the laboratory would be sufficient to account for the reduction.

In the well-known method of Fyfe and Schneider for the separation of arsenic from organic substances, depending upon the volatilization of the arsenic by boiling hydrochloric acid, no reducing agent was originally added, yet the arsenic present in the higher form of oxidation slowly distilled off. The organic matter itself served as reducing agent until Emil Fischer introduced the employment of ferrous chloride for that purpose.

Although I have not succeeded in obtaining from arsenic solutions a distillate absolutely free from arsenic—and it is almost impossible to devise any arrangement whereby every trace of organic matter, in the form of atmospheric dust, could be excluded in all stages of the operation—I believe I am justified in concluding that hydrochloric acid does not possess any reducing action *per se* upon arsenic compounds. Nevertheless, it is quite evident that it is not in practice a recommendable method to obtain arsenic-free hydrochloric acid, for the purpose of the Marsh-Berzelius test, from arsenical acid to which an oxidizing agent has been added. With reasonable care to exclude organic matter, only a minute fraction of the arsenic passes into the distillate. Thus Fresenius obtained from 10 grammes of sodium arsenate 4·4 milligrammes of sulphide, and I a much less but not determined quantity, from the arsenic acid produced 0·3 gramme arsenious acid. Hence, starting with a reasonably pure hydrochloric acid, it may be possible—and, after Mr. Allen's statement, we must take it that it *is* possible—to obtain in this way a distillate which fulfils the requirements of the method laid down by the Arsenic Committee; but my experiments clearly convince me that it is best to add a powerful reducing agent to drive off any arsenic there may be present, and to collect the distillate after such removal of the arsenic.

DISCUSSION.

Mr. ALLEN observed that Mr. Hehner's experiments showed strongly the necessity for the greatest care when these infinitesimal quantities of arsenic were being dealt with. Many conditions which were apt to be ignored and considered unimportant really played an important part in affecting the results. For himself, he could simply say that he had found, even in the case of hydrochloric acid containing considerable traces of arsenic, that the addition of a moderate excess of potassium permanganate and subsequent distillation yielded acid which, by the Marsh-Berzelius test, gave no indication of the presence of arsenic. But, after the experiences recorded by

Mr. Hehner, he would be quite prepared to find that, unless the conditions of success were discovered and rigidly adhered to, that method might break down as others had done.

Mr. E. H. JEFFERS said that, in the method of purification proposed by Dr. Thorne and himself—which was really a modification of the Reinsch process—one of the ordinary old-fashioned retorts was used, inclined upwards, the distillate being collected in a Wurtz flask placed with the side tube downwards, hence there were no organic connections. After the apparatus had been working for a few minutes the atmosphere inside the retort became practically free from organic matter. The specific gravity of the acid was, roughly, 1·100 to start with, and it practically continued to distil over at that gravity. The acid was supplied from a reservoir through a tube which passed down to the bottom of the retort, and round the lowest extremity of this tube a piece of copper gauze was placed. When once the solution was obtained colourless there was no further difficulty, the distillate being perfectly free from arsenic. The reducing action of the hydrochloric acid was shown by the fact that the acid previously extracted with copper gauze gave a dark-green solution on cooling overnight, whilst on heating the solution in the morning without copper its colour lightened considerably, and this must be due to the fact that the cupric chloride formed had already become partially reduced. It seemed to be of importance that the specific gravity of the acid should not vary from about 1·100.

Mr. HEHNER said that, if the arsenic was simply removed by distillation, he did not quite understand what bearing that would have upon his observations with regard to reduction, as the arsenic, after being boiled with the copper, would be already in the arsenious condition.

Mr. ALLEN said that since, after distillation, traces of arsenic were left behind in the acid, in presence of cuprous chloride it would be expected that such traces of arsenic, being in the arsenious state, would also come over on continued distillation.

Mr. JEFFERS said that the function of the copper was simply to keep the solution absolutely reduced. As soon as the solution boiled, the copper and the cupric chloride formed on cooling reacted, the dark-green solution becoming perfectly colourless. As the solution entered the retort it was reduced, and distillation was carried on so long as the liquid in the retort remained colourless. So long as the specific gravity of the solution did not exceed 1·100 no arsenic distilled over. If, however, the solution was thoroughly reduced to begin with, the copper would be blackened, owing to erosion and solution of the copper in the cupric chloride solution, and not to the deposition of arsenic.

THE USE OF BORIC ACID AND ITS SALTS AS FOOD PRESERVATIVES.

The following series of articles are freely translated from abstracts appearing in the *Zeit. für Untersuch. der Nahr. und Genussmittel*, 1902, v., 678-682 :

ON THE ACTION OF BORIC ACID AND BORAX ON HUMAN BEINGS AND ANIMALS. E. ROST. (*Arb. Kaiserl. Gesundh.-Amt.*, 1902, xix., 1-69.)—As the antiseptic action of boric acid is small, comparatively large quantities are necessary to preserve articles of food, it is quite possible for a person to take as much as 3 grammes daily of the preservative in his ordinary food. Meats, sausages, milk, butter, margarine, white and yolk of egg, fish, caviare, shellfish, etc., are frequently preserved by the addition of boric acid. The author found 3·87 per cent. in dry salt meat and 2·8 per cent. in shrimps. Boron compounds are stated to have no specific action on the enzymes of the stomach and intestines, except as regards their acid or alkaline properties. Borax retards to a small extent the coagulation of milk by rennet; the addition of borax to milk, especially when the latter is intended for infants' food, is therefore injurious. Large doses were found to cause local irritation and inflammation in dogs, cats, and rabbits, and also affected the action of the bowels. In two experiments on men it was found that doses of 1, 2, and 3 grammes of boric acid retarded the assimilation of albuminoids, the nitrogen contents of their urine being determined hourly before and after taking the boric acid. By taking the temperature of various dogs fed on borated meat, it was demonstrated that assimilation of the food was delayed. Experiments on other dogs showed that only large doses caused a loss of corpuscular albuminoids. It may be here mentioned that no essential difference was noticed in the action of boric acid and borax. A striking loss of weight of the animals was noticed. As this was not due to destruction of albumin or loss of water, it must be put down to oxidation of fat. Apparent increase in the digestion of albumin, shown when very large doses of borax were given, was due to the "salt" action of the borax, similar results being exhibited by large doses of common salt and potassium nitrate. A large consumption of water prevented these effects.

Assimilation experiments in the presence of boric acid were also carried out on four assistants. During a preliminary period of five to seventeen days the men were brought into a state of "nitrogen equilibrium," followed by administration of boric acid (3 grammes per diem) for twelve days. Two of the men then for a time received no boric acid, and afterwards underwent a second treatment. Finally, some days were devoted to studying the after-symptoms of the experiments. Two of the men showed a loss of weight due to loss of fat. The final observations also showed less secretion of urine and absorption of nitrogenous food materials. The two other assistants also showed a loss of weight. These two latter were also chosen for Rubner's experiments (see next paragraph), in which the amounts of expired carbon dioxide and water were determined. One of them diminished so suddenly in weight after taking 3 grammes of boric acid daily that the experiment had to be discontinued. The weight of the other also decreased, but increased when the boric acid was discontinued, and again fell when the latter was readministered. It was not demonstrated by the above experiments that boric acid affected the appetite. No influence upon health and appetite was noticeable.

Boric acid was not found by the author to influence the temperature, blood-pressure, or kidneys. As the elimination of boric acid by the urine takes from eight to fourteen days, its action is probably cumulative.

The author comes to the conclusion that the use of boron compounds in food should be forbidden.

THE ACTION OF BORIC ACID ON THE DIGESTION. RUBNER. (*Arb. Kaiserl.*

Gesundh.-Amt., 1902, xix., 70-88.)—According to the author, who comes to the conclusion after numerous experiments, boric acid has an important latent action on the digestive process. Not only the digestive organs themselves, but the whole alimentation is affected. The change produced, which may amount to a loss of 22 per cent. of energy and 30 per cent. of the utilization of nitrogen-free food, is a very important fact, and undoubtedly means injury to health, as the amount of fat in the body may be of the greatest importance, and the reduction of the fat must be followed by a rapid fall in albuminoids. Serious results may result in infant feeding, to invalids, old people, or convalescents by borated foods.

THE INFLUENCE OF BORAX ON METABOLISM IN MAN. R. O. NEUMANN. (*Arb. Kaiserl. Gesundh.-Amt.*, 1902, xix., 89-96.)—The experiments carried out by the writer on himself consisted of a preliminary period of four days, during which various observations were taken; then ten days with daily doses of 3 grammes of borax, followed by four days without borax, and concluding with daily doses of 5 grammes of borax for three days. During the first period nitrogen equilibrium existed; the secretion of nitrogen decreased during the first borax treatment, also in the intermediate four days, but was not further diminished by the larger doses of borax. His weight fell 1,200 grammes in seven days of the borax period. The flow of urine was somewhat increased, and boric acid could be detected for eighteen days after the last dose of borax had been taken.

THE INFLUENCE OF BORIC ACID ON THE UTILIZATION OF FOOD. A. HEFFTER. (*Arb. Kaiserl. Gesundh.-Amt.*, 1902, xix., 97-109.)—Four series of experiments were made, a considerable time apart. In each the author first fasted for eighteen to twenty hours, and then lived on milk (2 to 3 litres) and eggs (10) for forty-eight hours, afterwards again fasting for eighteen to twenty hours. In two of these periods no borax was taken, and in the other two the food was borated, one by taking 1 gramme of boric acid daily and in the second 4 grammes daily. In all the experimental periods the total nitrogen and dry solids were estimated in the fæces, as well as in the milk, and calculated in the case of the eggs. The boric acid was found to increase the solids and nitrogen in the excreta. When 1 gramme of boric acid was taken for two days, the dry solids amounted to 6·3 per cent. and the nitrogen 5·09 per cent. of the amount taken. By taking 4 grammes of boric acid in two days, the amounts were 8·8 per cent. and 8·9 per cent. respectively. During the time when no boric acid was taken the dry solids were 3·6 and 4·5 per cent., and the nitrogen 3·0 and 3·9 per cent. for the two days. The increase of the nitrogenous matter in the excreta was probably due to the diminished absorption of albuminoids as a result of the injurious effect of the boric acid on the mucous membrane of the intestines, boric acid often causing diarrhœa. The conclusion is that boric acid is not without objection when used as a preservative.

QUANTITATIVE ESTIMATIONS OF THE EXCRETION OF BORIC ACID FROM THE BODY. G. SONTAG. (*Arb. Kaiserl. Gesundh.-Amt.*, 1902, xix., 110-125.)—Single doses of 3 grammes of boric acid were administered to three healthy persons, and the boric acid then determined in the urine by titration in the presence of mannite after the urine had been freed from phosphates and neutralized. The accuracy of this method was tested by preliminary experiments. It was found that the urine was only free from boric acid after five, eight, and nine days had elapsed since taking the dose. About 50 per cent. of the boric acid was excreted in the first twelve hours, but the remainder very slowly. After the fifth day the quantity was so small that it could not be quantitively estimated. In all three experiments very similar results were obtained. It may be mentioned that in one case hourly variations were noticed, which were probably due to differences in the quantity of water secreted.

The Action of Rennet on Milk in the Presence of Boron Compounds and other Chemicals. A. Weitzel. (*Arb. Kaiserl. Gesundh.-Amt.*, 1902, xix., 126-166.)—Systematic experiments were made regarding the influence of ordinary preservatives and other compounds on the coagulation of milk by rennet. Aqueous solutions of the preservatives were employed when possible, but in the case of insoluble compounds the fine powder was added to the mixture of milk and rennet. Observations were taken as to whether the action of the rennet was accelerated or retarded by the chemicals used. The substances experimented with were divided into seven groups: (1) Alkaline: borax, sodium hydroxide, sodium carbonate, and sodium hydrogen carbonate. (2) Salts capable of precipitating lime: sodium oxalate, sodium fluoride, and sodium oleate. (3) Other salts having an alkaline reaction: sodium sulphite, salicylate, benzoate, propionate, acetate, and formate. (4) Neutral salts: sodium chloride, lithium chloride, sodium nitrate, perchlorate, tartrate, sulphate, ammonium sulphate, and magnesium sulphate. (5) Acid salts: sodium hydrogen tartrate, sodium hydrogen sulphate, and sodium persulphate. (6) Acids: boric, carbon dioxide, oxalic, benzoic, salicylic, protocatechuic, and gallic. (7) Formaldehyde, saccharin, and cane sugar.

The following results were obtained:

1. Borax retarded the coagulation when present in only small quantities (0·01 to 0·04 per cent.), and the amounts usually employed (1 gramme per litre of milk) stopped the action of the rennet altogether. All other alkaline salts acted similarly.

2. Coagulation was checked by those salts which precipitated the lime compounds. When the reaction became alkaline, the influence of alkalinity also showed itself.

3. The neutral salts generally had a retarding action. Some (sodium and lithium chloride) principally in concentrated solution, more feebly when present in small quantities. Magnesium sulphate in both concentrated and dilute solution had considerable influence.

4. Small quantities of the acids aided the coagulation. After carbon dioxide, boric acid had the most feeble action. The acid salts acted in the same manner as the acids.

5. The action of formaldehyde was so powerful that it must be considered as a direct poison to the rennet enzyme. Saccharin in small quantity had little influence, but stronger solutions greatly hindered the coagulation. Sugar, up to 20 per cent. of the weight of the milk, had no action.

The Amount of Boric Acid Present in Hams which have been Stored in Powdered Borax or Boric Acid. E. Polenske. (*Arb. Kaiserl. Gesundh.-Amt.*, 1902, xix., 167, 168.)—It is already known that fresh meat, when packed in borax, becomes thoroughly impregnated with the same. The author found the following quantities of borax in a piece of unsmoked ham weighing 600 grammes, which had been embedded in powdered borax for three weeks: in the fat, 0·076 per cent.; in the centre lean part, 2·73 per cent.; and in the outside lean portion, 4·05 per cent. An experiment was then made to show that boric acid and borax will also penetrate smoked hams. Two smoked hams, each weighing 5·5 kilogrammes, were packed—one in powdered borax and the other in powdered boric acid—and kept for four weeks in a dry place at the ordinary temperature. The hams were then well rinsed with water, and a section cut from each measuring 10 centimetres long, 6 centimetres wide, and 6 centimetres thick. These sections were then cut, beginning at the outside end, into small pieces of 0·5, 0·5, 1, 2, and 2 centimetres. The borax in these pieces, proceeding from the outside inwards, was then found to be 0·449, 0·137, 0·0879, 0·0764, and 0·0955 per cent. in the case of the ham packed in borax. The other ham gave 0·273, 0·173, 0·099, 0·099, and 0·0244 per cent. of boric acid.

W. P. S.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

The Cholin Test for Cognac. **H. Struve.** (*Zeit. anal. Chem.*, 1902, xli., 284-289.)—The products sold under the name of cognac are: (1) genuine cognac distilled from wine and matured in oak casks; (2) sweetened cognac, in which the crude spirit, often obtained from inferior wines, is mixed with sugar and caramel before storing; and (3) a fictitious article prepared by mixing a crude spirit with various essences and fruit flavours. According to the author, cholin does not occur in wine, genuine cognac, or other alcoholic liquids prepared by the fermentation process; and he has come to the conclusion that cognac in which cholin is present must be regarded as belonging to the third class. For the detection of cholin 50 c.c. of the cognac, from which the alcohol has been distilled, are digested for a few moments in a porcelain dish with a few drops of dilute sulphuric acid, then mixed with an excess of slaked lime or lead oxide and evaporated to dryness. The residue is extracted with 97 per cent. alcohol, and the alcoholic extract, which is usually colourless, evaporated to dryness. The residue is dissolved in a few drops of water, and a little of the liquid evaporated on a microscopic slide at 100° C. As a rule, a non-crystalline white residue is left, though white crystals of lead chloride are to be observed when lead oxide was used in the first evaporation. A drop of iodine solution is then added, a cover-glass placed over it, and the slide examined under the microscope. In the presence of cholin the characteristic needle-shaped crystals of cholin iodide will be observed in a few moments. These crystals at first increase in size, and then disappear after a short time. When the preparation is completely dry they can be again produced by the addition of more of the iodine reagent.

C. A. M.

Detection of Coal-Tar Colours in Preserved Fruits (Jams), Marmalade, etc. Reichelmann and Leuscher. (*Zeit. für öffentl. Chemie*, 1902, viii., 205.)—A portion of the jam is diluted with water and boiled for one hour, after adding a few white woollen threads. The latter are then taken out, washed with cold water, and treated with about 20 c.c. of 2 per cent. ammonia, no notice being taken of any colour change. At the end of fifteen minutes the threads are withdrawn, the liquid is made acid with sulphuric acid, and fresh wool is added. If this wool be appreciably dyed and the colour be unaltered on treatment with ammonia, coal-tar dyes are present, and may be approximately identified in the usual way. The boiled aqueous solution of the jam may be tested for the presence of saccharin and salicylic acid.

W. P. S.

Detection of Coal-Tar Colours in Pastry, Cakes, etc. Reichelmann and Leuscher. (*Zeit. für öffentl. Chemie*, 1902, viii., 204, 205.)—The following method is given for testing pastry and cakes which have already been coloured to some extent by the eggs employed in making them. About 50 grammes of the ground material are boiled for one hour under a reflux condenser with 75 c.c. of acetone and 10 c.c. of water. The solution is then poured off into another flask, the acetone is distilled off, 30 c.c. of water are added to the residue, and, after cooling, the solidified fat is removed by filtration. A few white woollen threads are added to the filtrate, which is then warmed for half an hour. Should tar colours be present the wool is dyed, and the colour may be identified by the usual tests. Water must be added to the acetone in order to extract all the dyes, and the acetone must afterwards be removed as completely as possible, so that all the fat shall separate out from the solution. The colouring-matter of the eggs remains in the fat. W. P. S.

Detection of Red Sanders Wood in Cocoa. Reichelmann and Leuscher. (*Zeit. für öffentl. Chemie*, 1902, viii., 203, 204.)—Sanders (sandal) wood may be detected in cocoa-powders, to which it is sometimes added for the purpose of masking the addition of starch, by microscopical examination, or by the following colour tests. From 2 to 3 grammes of the sample are shaken with about 10 c.c. of absolute alcohol. With pure cocoa the alcohol remains colourless, or is coloured only a faint yellow; the solution gives a white precipitate with sodium hydroxide, and no reaction with ferric chloride. The filtered alcoholic extract from sanders wood, or cocoa mixed with the latter, is, on the other hand, coloured, and gives, with dilute sodium hydroxide solution, an intense violet coloration. Should the wood have been previously exhausted, this coloration is less pronounced. Ferric chloride also yields a deep violet colour with the extract from unexhausted wood, but in the case of exhausted wood this reaction is only to be obtained by allowing a drop of ferric chloride solution to run on to the surface of the alcoholic extract. A violet ring is then formed, which disappears on shaking. Acetone may be used in place of alcohol for making the extraction. W. P. S.

Detection of Saccharin and Salicylic Acid in Foods. M. Spica. (*Gaz. chim. Ital.*, 1901, xxxi., [2], 41-46; through *Zeit. für Untersuch. der Nahr. und Genussmittel*, 1902, v., 620.)—The solution to be tested is shaken out with a mixture of ether and petroleum spirit, and the ethereal layer filtered through a dry filter. The filtrate is divided into three portions, which are placed in separate test-tubes and evaporated on the water-bath. The residue in one of the tubes is tested for salicylic acid by adding a few drops of concentrated nitric acid, warming the mixture, and dyeing wool with the resulting picric acid, if present. To another test-tube a little calcium oxide is added, and the tube heated until charring commences. The contents of the tube are then boiled with a few centimetres of water, and the solution is decanted into another tube containing a little hydrochloric acid and a small piece of zinc. After twenty minutes the solution is poured off, and a few drops of a solution of potassium nitrate and α-naphthylamine hydrochloride are added to it. Should saccharin have been present in the liquid under examination, a carmine red coloration is produced on standing for a few minutes. Extremely small traces of saccharin give a coloration after about an hour. The residue in the third test-tube is oxidized by the addition of a little sulphuric acid and potassium permanganate. After warming, any excess of permanganate is destroyed by means of oxalic or sulphurous acid, a little water is added, and a small quantity of a solution of diphenylamine hydrochloride is added to the bottom of the test-tube by a pipette. In the presence of saccharin a blue ring is formed at the junction of the two liquids.

W. P. S.

The Acetic Acid Test for Oil of Turpentine. P. W. Squire and C. M. Caines. (*Pharm. Journ.*, 1902, 512, 513.)—An acetic acid strictly complying with the British Pharmacopœia titration test (which requires the acid to be of 98·9 per cent. strength) does not form a clear solution with all samples of oil of turpentine when mixed in equal volumes. The acid must at least contain 99·5 per cent. of acetic acid to give clear solutions without subsequent turbidity. W. P. S.

Colour Reactions of Oil of Peppermint. P. Welmans. (*Pharm. Zeit.*, 1901, xlvi., 591, 592; through *Zeit. für Untersuch. der Nahr. und Genussmittel*, 1902, v., 633.)—P. Fiora has stated that oil of peppermint when mixed with phenol gives a greenish-blue coloration, which is destroyed by heating and reappears on cooling the mixture. Creosol, guaiacol, resorcinol, and sodium phenolate gave no reaction. The author finds that the reaction does not take place when absolutely pure phenol is used, but the addition of a drop of nitric acid free from chlorine causes the formation of a deep blue colour with a copper-coloured fluorescence. On warming, the colour changes to yellow. Chlorine, therefore, is not necessary to the reaction. The presence of acetic acid increases the intensity of the colour. Bromine and iodine give a violet coloration with the oil. The most pronounced blue colour is obtained by mixing 5 c.c. of oil of peppermint with 1 c.c. of iodine-mercuric chloride solution (25 grammes of iodine and 30 grammes of mercuric chloride dissolved in 1,000 c.c. of a mixture of acetic acid and acetic ether in equal parts). Mercuric chloride itself dissolves to a considerable extent in oil of peppermint, giving a green colour on gently warming. Further heating first gives rise to a violet-blue or deep green colour, according to the amount of mercuric chloride present; the blue or green colour then disappears and the whole of the mercury is precipitated as subchloride. The supernatant liquid is yellow, becoming green on standing and finally blue. In the presence of much mercuric chloride a deep green colour remains throughout the heating. Prolonged boiling with silver nitrate causes a mirror to be formed.

W. P. S.

TOXICOLOGICAL ANALYSIS.

Notes on the Serum Test for Blood in Forensic Cases. Ogier and Herscher. (*Ann. de Chim. anal.*, 1902, vii., 241-245.)—The authors confirm the statement of Linossier and Lemoine (ANALYST, xxvii., 250) that serum rendered specific for human blood-serum may often give precipitates with solutions of the blood-serum of other animals; but they point out that, if the amount of precipitate and the rapidity with which it appears be taken into account, a definite conclusion can be arrived at. Thus, in solutions of human blood-serum at 38° to 40° C. a flocculent precipitate appears throughout the liquid after about ten minutes, and within about thirty minutes the precipitation is nearly complete, and there is an abundant deposit at the bottom of the tube. On the other hand, in the case of other blood sera the formation of the precipitate does not begin until after a much longer time, usually several hours, and is invariably smaller. In testing suspected blood-stains the authors treat the stain with distilled water and place the solution in small tubes 10 to 12 centimetres in length, by 4 to 5 millimetres in diameter. At the same time they prepare solutions of approximately the same strength of the sera of the blood of man, and of five or six domestic animals. A few drops of the prepared serum reagent are then added to each tube, and the whole of the tubes shaken and immersed in a water-bath at 37° to 40° C. If after thirty minutes no precipitate is formed in the tubes containing the solution under examination, the authors conclude that it does not contain human blood, and take no notice of any precipitate that may subsequently be formed. If, on the other hand, a positive result be obtained, they report that it is extremely probable that the stain consisted of human blood. As it is not possible to carry out control tests with the blood of an indefinite number of animals, a more positive assertion is, in their opinion, not justifiable. With negative results, however, the case is different. Thus, in a recent criminal trial the prisoner asserted that a recent blood-stain was caused by the blood of a rabbit. The authors therefore prepared a serum specific for rabbit's blood-serum, and when this gave no precipitate within thirty minutes with a solution of the stain they were able to state with certainty that the stain did not consist of rabbit's blood. C. A. M.

ORGANIC ANALYSIS.

Reactions taking Place in the Determination of the Iodine Value. Harry Ingle. (*Journ. Soc. Chem. Ind.*, 1902, 587-595.)—As the result of a series of experiments, the author has come to the conclusion that the first action of the iodine solution is to add iodine monochloride directly on to the unsaturated bonds in the substance under examination. If in the iodochloride thus formed a carbon atom, to which the iodine or chlorine is attached, be joined also to a negative radical such as $.C_6H_5$ or .COOH, the iodochloride is not very stable. By the action of water it is decomposed, and the iodine or chlorine is replaced by hydroxyl, acid being at the same time produced. The acid may be estimated immediately after the determination of the iodine absorption by adding potassium iodate, and again titrating with thiosulphate. In the case of oils the quantity of hydriodic acid liberated enables one to ascertain approximately the amounts of fatty acid containing one and two unsaturated bonds respectively.

There is a tendency for iodochloride to be decomposed by the action of potassium iodide, the halogen being liberated again. The iodine value may thus appear too low. It is desirable to use sufficient chloroform to retain the whole of the iodochloride in solution, and to add water before introducing the potassium iodide solution. Some of the iodochloride is thus converted into a hydroxyl compound, which is more soluble in the chloroform, and is not so readily decomposed by the potassium iodide.

Waller's solution contains iodine hydrochloride—ICl, HCl—and as this has to be decomposed before addition takes place, it is not only slower in action than Hübl's solution, but also may, on this account, with certain substances yield either no iodine value at all, or one much below the correct amount. This is especially the case where an adjacent negative radical exerts a protecting influence over the unsaturated bonds.

Substitution does not take place during the determination of the iodine value by the methods ordinarily employed. A. M.

Determination of Pentanes and Pentosans. E. Kröber and C. Rimbach; communicated by G. B. Tollens. (*Zeits. f. angew. Chem.*, 1902, xv., 477.)—Compare Analyst, 1899, 178, and 1901, 133. Rimbach has distilled arabinose and xylose with hydrochloric acid of different strengths to ascertain whether the yield of furfurol-phloroglucide is thereby affected. The maximum yield was obtained with acid of specific gravity 1·08 to 1·10, but he recommends that 1·06 be still adhered to, as it is the generally-accepted strength. The distillation is carried out in a metal bath with the constant addition of small quantities of acid, until the distillate no longer gives a red coloration with aniline acetate. More than the calculated quantity of phloroglucinol is dissolved in hydrochloric acid (specific gravity 1·06) and added to the distillate, which is then made up to 400 c.c. and allowed to stand twelve to fourteen hours. The phoroglucide is collected in a porcelain Gooch crucible, using slight suction. It is washed with 150 c.c. of water, taking care to keep the precipitate covered with liquid. It is then dried for four hours in a water-oven, transferred to a stoppered tube, allowed to cool, and weighed. To the weight found it is necessary to add ·0052 gramme to compensate for phloroglucide dissolved in the acid and wash-water. The corrected weight may be then calculated to furfurol or arabinose, etc. The reaction between furfurol and phloroglucinol takes place in accordance with the equation—

$$C_5H_4O_2 + C_6H_6O_3 = C_{11}H_6O_3 + 2H_2O.$$

More accurate results are obtained by use of Kröber's tables, which are based on a large number of experiments with different pure materials. The following are a few of the figures:

Phloroglucide.	Furfurol.	Arabinose.	Araban.	Xylose.	Xylan.	Pentose.	Pentosan.
0·030	0·0182	0·0391	0·0344	0·0324	0·0285	0·0385	0·0315
0·050	0·0286	0·0611	0·0538	0·0507	0·0446	0·0559	0·0492
0·074	0·0411	0·0875	0·0770	0·0726	0·0639	0·0801	0·0700
0·100	0·0546	0·1161	0·1022	0·0964	0·0848	0·1063	0·0935
0·169	0·0904	0·1919	0·1688	0·1592	0·1401	0·1756	0·1546
0·200	0·1065	0·2250	0·1984	0·1874	0·1649	0·2065	0·1817
0·300	0·1581	0·3335	0·2935	0·2784	0·2450	0·3060	0·2693

The last two columns are for use in cases where it is not known which pentose or pentosan is present. In most plant substances araban and xylan occur together, but gums contain araban almost exclusively, and wood and straw only xylan.

A. M.

The Initial Action of Iodine and other Oxidizing Agents in the Hydrolysis of Starch and Dextrin. F. E. Hale. (*Zeits. Anorg. Chem.*, xxxi., 100.)—As the result of an elaborate investigation, the author comes to the conclusion that the small loss of iodine, as well as the production of red or purple colorations, which are occasionally met with when arsenious or antimonious oxide is titrated with iodine solution, starch being used as indicator, is probably due to the presence of soluble starch in the starch. A small quantity of this substance appears to be oxidized by the iodine, as well as by hydrogen peroxide or potassium permanganate in alkaline solutions, and the resulting organic acid exerts a hydrolysing action on the remainder with the formation of erythrodextrin, which gives a red colour with iodine. The presence of arsenious or antimonious oxide apparently favours the reaction.

A. G. L.

On Malt Diastase. A. R. Ling and B. F. Davis. (*Journ. Fed. Institut. Brewing*, 1902, viii., 473-495.)—As the result of numerous experiments on the nature of the diastase in different malts used for brewing purposes, the following conclusions are arrived at: Diastase prepared from well-grown, low-dried malt hydrolyses the starch in a 2 to 5 per cent. starch-paste at temperatures between 50° and 60° C. to a point at which the specific rotatory power and the cupric reducing power of the matter in solution, after the hydrolysis has proceeded for about one and a half hours, are:

$$[\alpha]_{D3\cdot93}\ 149\cdot5 - 150. \quad R_{3\cdot93}\ 77 - 78.$$

At the end of twenty-four hours these constants are practically those of maltose, and in the solution nothing but maltose could be detected. On the other hand, diastase prepared from malt grown under abnormal conditions (*e.g.*, made in the laboratory) does not, even when used in large excess, hydrolyse starch completely to maltose. The nature of the product other than maltose has not yet been determined. A similar effect to the latter is also apparently produced by the action on starch of diastase which has been slowly heated to 115·5° to 120° C.; but diastase which has been rapidly heated to this temperature produces a more pronounced effect. The final temperature at which malt has been kilned is not alone a criterion of its behaviour towards starch, nor is the "diastatic power" as determined by Lintner's method. When diastase is heated in solution to above 65° C. its action on starch paste appears to be quite different to that of diastase solution which has not been heated higher than 60° C. This is indicated by the constants of the matter in solution, and also by the fact that dextrose is invariably present as one of the final products of the hydrolysis. W. P. S.

The Determination of Albuminoid and Proteid Ammonia. L. W. Winkler. (*Zeit. anal. Chem.*, 1902, xli., 290-300.)—According to the author, an estimate of the nitrogenous organic impurities in water can be formed in a much simpler and more certain manner by oxidation with potassium persulphate in acid solution than by the ordinary method of oxidation with alkaline permanganate. Since the amount of ammonia thus split off and determined directly in the liquid without distillation differs from that obtained as albuminoid ammonia, the author distinguishes it by the name of "proteid ammonia." Preliminary experiments with solutions of caffeine and other nitrogenous compounds having shown that there was only a trifling increase in the amount of proteid ammonia after fifteen minutes' heating, that limit of time was adopted in all subsequent determinations. The following table gives the comparative amounts of proteid and albuminoid ammonia obtained from different compounds together with the theoretical quantities:

Substance.	Proteid Ammonia.	Albuminoid Ammonia.	Ammonia Theoretically Obtainable.
	Per Cent.	Per Cent.	Per Cent.
Urea	27·0	2·2	56·67
Hippuric acid	7·4	3·1	9·45
Uric acid	10·6	4·3	40·17
Caffeine	15·2	15·0	26·29
Leucin	10·2	11·3	12·98
Tyrosin	7·9	8·0	10·30
Aspartic acid	11·2	11·8	12·81
Gelatin (18·3 per cent. N.)	9·1	8·2	22·22
Albumin (15·7 per cent. N.)	7·1	6·7	19 06

The solutions required in the determination of proteid ammonia are: (1) Potassium persulphate 1 per cent. solution. In order to free the commercial salt from ammonium persulphate, 15 grammes are powdered and dissolved in 100 c.c. of water at 60° C., to which is added 1·5 grammes of potassium hydroxide, and the solution filtered through cotton-wool and left for some hours in a cool place. The crystals are collected, washed with cold water, and dried at the ordinary temperature. The solution of the salt is not very stable, but is applicable so long as barium chloride does not produce more than a turbidity. (2) An approximately $\frac{1}{5}$ N. solution of sulphuric acid, prepared by diluting 6 c.c. of pure concentrated acid to a litre. (3) Ammonium chloride solution, containing 0·315 gramme per litre; 1 c.c. = 0·1 milligramme of ammonia. (4) Nessler's reagent, prepared by triturating 10 grammes of mercuric iodide with water, adding 5 grammes of potassium iodide, and mixing the liquid with a completely cold aqueous solution of 20 grammes of sodium hydroxide, the total amount of water used being 100 c.c. Mercuric chloride must not be used for the reagent. (5) Sodium potassium tartrate solution, containing 50 grammes in 100 c.c., with 5 c.c. of Nessler's reagent as a preservative. In the determination of the ammonia equal parts of solutions 4 and 5 are mixed, and termed the "mixed reagent." If any separation of mercuric iodide occur in using this mixed reagent, 0·1 to 0·2 gramme of potassium iodide should be dissolved in 50 c.c. thereof.

In the determination of the proteid ammonia, 100 c.c. of the water under examination are mixed with 5 c.c. of the sulphuric acid solution and 5 c.c. of the potassium persulphate solution, and if the liquid has not then an acid reaction, an additional 5 c.c. of the acid are added. The flask is covered with an inverted beaker and placed on a water-bath in which the water is boiling vigorously, so that the contents of the flask reach a temperature of 90° C. in four to five minutes, and of 95° C. after two or three minutes longer. After fifteen minutes the flask is cooled in a current of water and its contents transferred to a cylinder, into which is also introduced, drop by drop, 5 c.c. of the mixed reagent. At the same time 100 c.c. of the water under examination are placed in a duplicate cylinder and mixed with 5 c.c. (or 10 c.c.) of the sulphuric acid, then with 5 c.c. of the mixed reagent, added drop by drop, and then with 5 c.c. of potassium persulphate solution. The yellow colour of the liquid in the second cylinder will be much fainter than that of the first cylinder. The standard ammonium chloride solution is now added until the colours of the liquids in both cylinders are the same, and the number of c.c. required gives the amount of proteid ammonia in the water.

From the results of experiments on different waters, the author states that in absolutely pure natural water no proteid ammonia is found. He considers that if the proteid ammonia amounts to more than 0·1 milligramme per litre, the water is defective from a hygienic point of view for drinking purposes. Since the method only measures the soluble nitrogenous impurities, the albuminoid ammonia determination is preferable in the case of strongly turbid water and also for coloured waters.

The spontaneous purification of impure water exposed to the air is shown in the following series of determinations. The tap-water before the addition of urine (1 c.c. to 10 litres) contained in 1 litre no free ammonia, 0·05 milligramme of proteid ammonia, no nitrous acid, and 1·6 milligrammes of nitric acid, and required 3·09 milligrammes of potassium permanganate in the oxygen absorption.

Water Containing Urine.	Free Ammonia.	Proteid Ammonia.	N_2O_3.	N_2O_5.	$KMnO_4$ Consumed.
	Milli-grammes.	Milli-grammes.	Milli-grammes.	Milli-grammes.	Milli-grammes.
Freshly mixed	0·19	0·75	0·00	1·6	5·08
After 1 week	0·20	0·80	0·00	1·7	4·61
,, 2 weeks	0·00	0·77	0·38	2·1	3·91
,, 4 ,, ...	0·00	0·76	0·00	2·7	3·91
,, 6 ,, ...	0·00	0·10	0·00	7·4	3·79
,, 8 ,, ...	0·00	0·09	0·00	10·0	3·06
,, 10 ,, ...	0·00	0·08	0·00	8·5	2·99
,, 12 ,, ...	0·00	0·07	0·00	7·2	2·99

C. A. M.

Examination of Glue and Gelatine. A. Müller. (*Zeits. f. angew. Chem.*, 1902, xv., 482.)—The sample is dissolved in water, and the glutin is precipitated by the addition of a known quantity of tannic acid, the excess of tannic acid being estimated by titration with permanganate. Ten grammes of the sample are allowed to swell in water overnight, then heated and made up to 500 c.c. Quantities of 10 c.c. are taken, and tannin solution (5 grammes in 1 litre) is added gradually, with constant stirring—30 c.c. in the case of leather-glue or gelatine, and 25 c.c. in the case of bone-glue—then 20 c.c. of a 5 per cent. solution of potash alum are added, the liquid is stirred on the water-bath for a minute, then rapidly filtered and washed with water at 30° C. The filtrate is titrated with standard permanganate solution. In order to correct for reducing substances in the glue not precipitated by tannin a second determination is made, in which the filtrate is treated with hide powder and then titrated. The difference between the two gives a measure of the glutin. Under the conditions laid down, 100 grammes of tannin precipitate 139·1 of glutin. The results agree well with those obtained by Kjeldahl's process, but, unlike the latter, they are unaffected by the presence of nitrogenous impurities. A. M.

Estimation of Guanidine. A. Vozárik. (*Zeit. für angew. Chem.*, 1902, xv., 670-672.)—The method is based upon the precipitation of guanidine by picric acid. As the compound formed—$CH_5N_3.OHC_6H_2(NO_2)_3$—is soluble at the ordinary temperature in about 1,280 parts of water, the precipitation is carried out in an ammoniacal solution of ammonium picrate previously saturated with guanidine picrate. This solution is prepared by dissolving 8 grammes of ammonium picrate, 0·075 gramme of guanidine picrate, and 5 c.c. of ammonia (specific gravity 0·91) in 1,000 c.c. of water. In carrying out the process, 8 grammes of the guanidine salt to be tested are dissolved in water which has been rendered ammoniacal, and made up to 1 litre. It is essential that the strength of this solution as regards its guanidine salt contents should be from 0·8 to 1 per cent. After allowing to stand for two to three hours in order that any lead present may settle, 25 c.c. are precipitated by dropping in 100 c.c. of the ammonium picrate solution. The precipitate is filtered after standing six to twelve hours through a Gooch's crucible containing a little asbestos, washed with ammonium picrate solution (the latter being removed as much as possible by suction), dried at 110° C., and weighed. The precipitate retains about 1 per cent. of its weight of the ammonium picrate solution, and the asbestos 2·5 per cent. of its weight. The weight of the precipitate is corrected for these amounts.

W. P. S.

To Distinguish the different Varieties of Carbon. E. Donath and B. Margosches. (*Chem. Ind.*, 1902, xxv., 226.)—In consequence of the use of different modifications of carbon for the manufacture of arc-light pencils and electrodes, it is of importance to be able to distinguish them. One is able to do this to some extent by taking advantage of their different behaviour to oxidizing agents. Diamonds are not attacked by a mixture of potassium chlorate and concentrated nitric acid. They burn in oxygen to carbon dioxide at temperatures above 700°. They are rapidly attacked by fused alkaline carbonates.

Graphite, on treatment with fuming nitric acid and potassium chlorate, is oxidized to graphitic acid. Some varieties of natural graphite, when soaked in a little nitric acid and then ignited, swell up with the formation of worm-like excrescences. Those varieties that do not give this reaction have been termed "graphitite" by Luzi. Graphite is not attacked by nitric acid (1·4), and only slowly by a mixture of this with concentrated sulphuric acid.

Graphite, retort carbon, coke, and petroleum coke are not dissolved by boiling nitric acid (1·4), but are attacked somewhat by a mixture of this with concentrated sulphuric acid. Anthracite, coal, brown coal, charcoal, sugar charcoal, soot, and pure amorphous carbon are partially dissolved by the nitric acid with the formation of an intense brownish-red coloration. In the mixture of nitric and sulphuric acids solution is more rapid.

By boiling with concentrated alkaline permanganate solution coal and brown coal are oxidized to oxalic acid. Graphite, on the other hand, is not attacked, and coke and charcoal only to a small extent. Brown coal, lignite, and pitch may be distinguished from coal by treating with brominated potash. The former dissolves readily, but the latter is little affected. Charcoal also is but little attacked.

Graphite and coke can be differentiated by fusion with sodium sulphate. The former is unaffected, but the latter reduces the sulphate to sulphide and sulphite.

A characteristic property of charcoal is that, on continued heating with concentrated sulphuric acid, it dissolves with the formation of sulphurous acid and brown coloration. Coal under the same conditions is not entirely dissolved.

Ordinary soot and lamp-black may be distinguished from soot made from naphthalene by extracting with petroleum ether. The latter yields a fluorescent solution, which on evaporation leaves a residue of characteristic odour.

Valuable information may often be obtained by submitting a sample to dry distillation, or by examining for impurities, such as nitrogen and sulphur. Microscopic examination, combined with treatment with chromic acid, is also often useful.

The amount of anthracite in an adulterated graphite may be roughly estimated by observing the coloration produced on boiling with nitric acid, and the quantity of oxalic acid produced by treatment with alkaline permanganate. Charcoal may be detected in the presence of coke by the coloration with nitric acid. Retort carbon is distinguished from graphite by not giving Brodie's graphitic acid reaction. Electric graphite can be distinguished from natural only by its physical properties. A. M.

INORGANIC ANALYSIS.

The Determination of Copper as Cuprous Thiocyanate in the Presence of Bismuth, Antimony, Tin, and Arsenic. R. G. van Name. (*Zeits. Anorg. Chem.*, xxxi., 92.)—The author finds that the separation of copper from bismuth by precipitation as thiocyanate is not very satisfactory, even in the presence of both hydrochloric and tartaric acids, as the presence of much free acid exerts a solvent action on the precipitate, whilst if too little acid is present the latter will be contaminated with basic salts of bismuth. He finds it best to start with a fairly acid solution of the two metals, and roughly ascertain, by means of a few blank tests on separate portions of the solution, the maximum quantity of ammonium bisulphite solution which may be added (to neutralize the acid) without precipitating bismuth. From antimony and tin, on the other hand, the separation is easily effected in the presence of small quantities only of hydrochloric and tartaric acids, whilst for the separation from arsenic the presence of a fair amount of hydrochloric acid is all that is necessary. A. G. L.

On a Modification of Rose's Method of Separating Nickel and Cobalt. R. L. Taylor. (*Chem. News*, lxxxv., 269.)—In Rose's method the cobalt is precipitated as sesquioxide from dilute solutions containing free hydrochloric acid by means of bromine or chlorine, and barium, strontium or calcium carbonate, nickel remaining in solution. The process, even as modified by Henry (*Chem. Gazz.*, 1855, 237), requires several hours, and has consequently fallen into disuse. The author of the present paper found, however, that if neutral solutions are used instead of acid ones, the method not only becomes more accurate, but the precipitation of the cobalt is complete in a few minutes. From his experiments, he comes to the conclusion that the retarding effect of the free acid on the precipitation of cobalt is due to the production of considerable quantities of free carbonic acid. The method, as recommended by him for both quantitative and qualitative analysis, consists simply in adding to the cold neutral solution of the metallic chlorides a paste of precipitated barium carbonate and water and then bromine water, allowing to stand for ten minutes, and filtering the precipitate off. If the solution is allowed to stand for more than an hour in contact with air some nickel may be precipitated. If the solution of the chlorides contains free acid, this must be removed by evaporation, neutralizing with a caustic alkali, or else by adding excess of barium carbonate, boiling to expel the free carbonic acid, and cooling before adding the bromine water.

A. G. L.

The Volumetric Determination of the Double Phosphates of Ammonium with Cadmium, Cobalt, Manganese, and Zinc. H. D. Dakin. (*Zeit. anal. Chem.*, 1902, xli., 279-284.)—The method consists of dissolving the precipitate of the double phosphate in an excess of normal acid, and titrating the excess with normal alkali, using methyl orange as indicator, as in Stolba's method of titrating ammonium magnesium phosphate. The end-point of the titration is reached when the metal present is converted into sulphate or chloride and the phosphoric acid into monophosphate. Thus :

$$M''(NH_4)PO_4 + H_2SO_4 = M''SO_4 + (NH_4)H_2PO_4.$$

Cadmium ammonium phosphate precipitated in the cold and dried at 100° C. contains 1 molecule of combined water. In test experiments the amounts of cadmium found were within 0·0007 gramme of the theoretical amounts. The conditions for the quantitative separation of cobalt, manganese, and zinc as double phosphates with ammonium were described in a former communication (*Zeit. anal. Chem.*, xxxix., 273 and 784). In each case satisfactory results were obtained in the test experiments. (*Cf.* Walker, ANALYST, xxvi., 304.) C. A. M.

The Detection of Traces of Vanadium, and the Separation of Vanadium from Molybdenum. P. Truchot. (*Ann. de Chim. anal.*, 1902, vii., 167, 168.)—In the author's electrolytic method of determining vanadium, the deposit on the cathode is brownish-yellow and more or less iridescent. On washing the deposit with water and alcohol, evaporating the latter by gentle heat, and treating the deposit with a little potassium bisulphate and 2 to 3 drops of sulphuric acid, a characteristic yellow coloration is obtained.

On now adding a few crystals of strychnine sulphate, a bluish-violet colour changing to rose is produced.

This reaction is capable of detecting 1 part of vanadic acid in 100,000.

The colorations given by other alkaloids and by phenols with vanadic anhydride are as follows : Atropine, nil ; caffeine, nil ; santonin, nil ; phenol, dark green ; and pyrogallol, very dark brown.

Separation of Vanadium from Molybdenum.—Molybdenum is deposited as a black hydroxide upon the cathode under exactly the same conditions as described above in the case of vanadium. On ignition, this hydroxide is converted into molybdic acid, which is completely volatilized at a red heat.

When both vanadium and molybdenum are present, the deposit is first ignited gently and then strongly until the weight becomes constant.

As a rule, the ignition is complete in about thirty minutes when the mixture of the two oxides is not more than 0·1 gramme. C. A. M.

The Determination of Vanadium. H. Cormimbœuf. (*Ann. de Chim. anal.*, 1902, vii., 258-260.)—The following simple modification of Roscoe's method is stated to be more accurate than other gravimetric methods, and to take only twenty-four hours : The vanadium compound is converted into potassium or sodium vanadate, which is dissolved in water, and filtered from the insoluble oxides of iron, magnesium etc. The filtrate is acidified with acetic acid and a sufficient quantity of lead acetate added. Under these conditions the vanadic acid is completely precipitated, although the lead vanadate is not always constant in composition. The precipitate is collected on a weighed filter, washed with water containing a little acetic acid, dried at 100° C. and weighed. It is then detached from the paper and pulverized in a mortar. A weighed portion of the powder is dissolved in water acidulated with nitric acid, and the lead precipitated by adding a slight excess of concentrated sulphuric acid. The liquid is slightly diluted with water and filtered from the lead sulphate, and the filtrate evaporated to dryness in a weighed porcelain crucible on the water-bath. The temperature is then gradually raised until, finally, it reaches red heat, at which stage the vanadic acid is partially fused and quite pure. The crucible is then cooled and weighed, and the total amount of vanadic acid in the lead vanadate calculated from the weight thus obtained from the aliquot portion. C. A. M.

The Determination of Thallium in the Thallous State. V. Thomas. (*Bull. Soc. Chim.*, 1902, xxvii., 470-471.)—The author's method is based upon the oxidation of the thallous salt by means of gold bromide, and the gravimetric determination of the metallic gold—

$$3TlCl + 2AuBr_3 = 3TlClBr_2 + 2Au.$$

About 5 grammes of the salt (chloride) are dissolved in hot water, and the solution heated on the water-bath and treated with a solution of gold bromide in sufficient quantity. The whole is then kept hot for eight or ten hours, after which the gold is collected on a filter, dried, and weighed. The weight of gold multiplied by the factor, 1·5533, gives the weight of thallium. In this way the author obtained five results ranging from 85·02 to 85·30 per cent. of thallium, the theoretical amount in thallous chloride being 85·21 per cent. The gold bromide solution is prepared by dissolving gold in hydrobromic acid containing bromine, and evaporating the solution to dryness on the water-bath. The crystals (bromo-auric acid) are dissolved in water, and the solution filtered before use if necessary. C. A. M.

The Separation of Beryllium. G. Wyrouboff. (*Bull. Soc. Chim.*, 1902, xxvii., 733, 734.)—There is considerable difficulty in effecting a quantitative separation of beryllium from iron and aluminium, with which it is invariably associated in the emerald. In examining various compounds of beryllium, the author has found that the oxalate, $Be_2O_3(C_2O_3)_6.3K_2O$, is only sparingly soluble, 100 parts of water dissolving 1·4 parts of the salt at 15° C., whilst the corresponding oxalates of iron, aluminium, and chromium are soluble in two or three times their weight of water. In this way it is possible to effect a rapid and approximately complete separation of beryllium. The mineral is attacked with potassium hydroxide, the silica removed in the usual way, and the solution of chlorides evaporated to a small volume and treated with a concentrated solution of potassium hydrogen oxalate. The crystalline precipitate, which is soon deposited, is washed with water, and is quite free from iron or aluminium. C. A. M.

A Colour Reaction of Uranium Salts and Hydrogen Peroxide. J. Aloy. (*Bull. Soc. Chim.*, 1902, xxvii., 734, 735.)—On treating a salt of uranium with hydrogen peroxide and then with potassium carbonate in either the solid form, or in a concentrated solution, a red coloration is produced, and on then adding to the liquid two or three times its volume of alcohol a red precipitate is rapidly deposited. In applying this test to the detection of traces of hydrogen peroxide, it is best to make a solution of uranium nitrate in 95 per cent. alcohol, then to add a few drops of the liquid under examination, and, lastly, a few crystals of potassium carbonate. The reaction takes place in two stages, a white peruranic hydroxide being formed by the action of the hydrogen peroxide, and subsequently decomposed by the alkali carbonate. The red precipitate is very unstable, rapidly losing part of its oxygen at the ordinary temperature.

C. A. M.

Aromatic Bases as Precipitants for Rare Earth Metals. **Alice MacMichael Jefferson.** (*Journ. Amer. Chem. Soc.*, xxiv., 540.)—The behaviour of six of the rare earth metals with aromatic bases was examined, and it was found that zirconium is precipitated quantitatively in the form of double salts by aniline, o-toluidine, xylidine, dimethyl- and diethyl-aniline, benzylamine, pyridine, piperidine, and quinoline. Thorium is precipitated by aniline, o-toluidine, xylidine, dimethyl-aniline, diethyl-aniline, pyridine, piperidine, and quinoline. Cerium is completely precipitated by aniline, o-toluidine, diethyl-aniline, benzylamine, pyridine, and piperidine, whilst lanthanum, neodymium, and praseodymium are only precipitated by benzylamine and piperidine.

Fairly good separations can consequently be effected by quinoline of thorium and zirconium from neodymium, and by aniline of thorium and zirconium from lanthanum; lanthanum and thorium may also be separated by means of quinoline. O-toluidine separates cerium from praseodymium; thorium and praseodymium may be separated by aniline, and lanthanum and praseodymium from thorium by means of xylidine. In most cases it is advantageous to repeat the separations.

A. G. L.

Analyses required for an Electrolytic Alkali Works. **J. H. James and J. C. Ritchey.** (*Journ. Amer. Chem. Soc.*, xxiv., 469.)—The free chlorine, sodium hypochlorite, and sodium chlorate in the spent brine are determined as follows: For free chlorine, 50 c.c. of the brine are treated with 10 c.c. of a saturated solution of potassium iodide for five minutes, and the liberated iodine titrated with decinormal thiosulphate solution. For sodium hypochlorite, 50 c.c. of the brine are titrated with arsenious acid, using drops of starch and potassium iodide solution on a plate as indicator. The hypochlorite is obtained by subtracting from the value found the equivalent of the chlorine found above. For sodium chlorate, 50 c.c. are placed in an 8-ounce flask, the quantity of arsenious acid solution found above to be equivalent to the free chlorine plus sodium hypochlorite is added, and then a weighed amount of ammonium ferrous sulphate in excess of the chlorate present. The flask is closed by a small funnel, heated, and 15 c.c. concentrated sulphuric acid are slowly added, 1 c.c. at a time, after which the solution is boiled gently for three or four minutes. After cooling, the excess of ferrous salt is titrated with bichromate solution. The quantity of ferrous salt oxidized corresponds to the chlorate present.

The quantity of water in the bleaching powder is determined by heating a mixture of 1 gramme of the sample with 5 grammes of copper oxide in a porcelain boat placed in a porcelain tube. The water driven off is absorbed in a sulphuric acid tube and weighed. Between the boat and the absorption tube a mixture of scrap sheet and cement silver is placed to absorb any chlorine escaping from the boat. The tube is heated gently at first, the portion under the boat being finally raised to the hottest temperature obtainable, and kept at this temperature for one hour and a half. A current of dry air is drawn through the tube during the heating. The reaction in the boat occurs according to the equation:

$$Ca(OCl)_2 + 4CuO = CaCl_2 + 2O_2 + 2Cu_2O.$$

A. G. L.

The Microchemical Detection of Alkalies and Acids. F. Emich. (*Monatsh. für Chem.*, 1901, xxii., 670-678; and 1902, xxiii., 76-80.)—*Microchemical Detection of Acids and Alkalies by means of "Litmus Silk."*—Silk dyed with a purified solution of litmus is recommended as a suitable reagent. The red silk is prepared by boiling the silk with a solution prepared by extracting commercial litmus with water, rejecting the first extract, boiling the residue with a fresh portion of water, filtering the solution, and adding sulphuric acid in excess to the boiling filtrate. After thirty minutes the dyed silk, which has a violet-red colour, is washed in a current of water, dried, and kept in the dark.

The blue silk is prepared by soaking the red silk in water rendered faintly alkaline, washing it rapidly, pressing it between filter-paper, and drying.

In using the litmus silk, a single cocoon fibre is fixed to a fragment of wax and cut with sharp scissors so as to leave about a centimetre free. This is purified with a drop of alcohol, and examined under the microscope to insure absence of impurities. A droplet (about 0·05 milligramme) of the liquid is then placed on a suitable surface, and the end of the silk introduced, and after the evaporation of the liquid again examined under the microscope.

The smallest quantities of an alkali or acid are capable of detection by this method.

C. A. M.

Common Errors in the Determination of Silica. W. F. Hillebrand. (*Journ. Amer. Chem. Soc.*, xxiv., 362.)—The author calls attention to the known fact that at least two evaporations, alternating with two filtrations, are absolutely necessary in order to render silica insoluble. He also shows that the generally accepted view that the silica in the filtrate is completely precipitated by ammonia or sodium acetate, if sufficient iron or aluminium is present, is incorrect, and that the silica contained in the oxides of iron and aluminium is only partly left undissolved when the oxides are fused with potassium pyrosulphate and the melt treated with water or acid, since silica is appreciably soluble in fused potassium pyrosulphate. The statement of

Lunge and Millberg (*Zeits. angew. Chem.*, 1897, 425), that silica can be rend completely anhydrous merely by ignition over a Bunsen burner, is found by the aut to be correct as regards the silica obtained by the action of water on silicon tet fluoride, but does not apply to silica separated by acids from silicates, for which prolonged use of the blow-pipe is necessary. A. G. L

A New Volumetric Method for the Determination of Free and Combined Sulphuric Acid. W. Müller. (*Berichte*, 1902, xxxv., 1587-89.)—This is based upon the fact that sulphuric acid is precipitated by benzidene hydrochloride. The solubility of benzinide sulphate, according to the author's experiments, at 25° C is about 0·02 per cent., and this is diminished both by the addition of su acid and of benzidene salts. It is, moreover, possible, on the other hand, to the combined acid in a benzidene salt with standard alkali, phenolphthale used as indicator, and thus to standardize the benzidene hydrochloride solutic

By using a measured excess of benzidene hydrochloride and determin amount left in the filtrate from the precipitate, the difference corresponds amount of sulphuric acid. Since benzidene sulphate mechanically carries little benzidene hydrochloride from a cold solution, the precipitation should b from a hot solution. A definite quantity of the sample under examination is tit with alkali, using phenolphthalein as indicator, and the neutralized solution dilute to 150 c.c. and warmed on the water-bath. A measured excess of 20 to 30 per cent. of benzidene solution beyond the amount required to effect complete precipitation is then added, and the flask kept on the water-bath for a few minutes until the benzidene sulphate separates. The liquid is then cooled, diluted to 250 c.c., and filtered, and an aliquot portion of the filtrate titrated back with standard alkali. The difference between the amount of alkali required by the benzidene hydrochloride used and that now consumed gives the amount of sulphuric acid. C. A. M.

The Detection of Nitric Acid in the Presence of an Alkali-metal Ferrocyanide or Ferricyanide. A. F. Leuba. (*Ann. de Chim. anal.*, 1902, vii., 258.)—The test for nitric acid by the addition of sulphuric acid and a ferrous salt to the solution cannot be made in the usual manner in the presence of alkali-metal ferrocyanides or ferricyanides, since these form dark-blue compounds. To obviate this, a slight excess of a concentrated solution of cadmium chloride is added, the liquid filtered from the insoluble cadmium ferro- or ferri-cyanide, and the test for nitric acid applied to the filtrate. C. A. M.

On the Influence of Potassium Ferrocyanide on the Precipitation of Phosphoric Acid. A. F. Leuba. (*Ann. de Chim. anal.*, 1902, vii., 257.)—Potassium ferrocyanide interferes with the precipitation of phosphoric acid by ammonium molybdate, owing to its yielding a dense reddish-brown precipitate, to which has been assigned the formula $Mo(OH)_4$. This precipitate is soluble in an excess of hot nitric acid, forming a brown solution, from which, however, the phospho-molybdate still does not separate. On the other hand, the brown precipitate readily dissolves in sodium carbonate solution, and on exactly neutralizing the liquid with nitric acid and heating it very slightly the yellow phospho-molybdic compound separates out.

C. A. M.

Iodometric Determination of Ferro- and Ferri-cyanides. E. Rupp and A. Schiedt. (*Berichte*, vol. xxxv., p. 2430.)—In De Haen's method for the titration of ferrocyanides with permanganate the end-point is indistinct, and the results are, as a rule, from 1 to 3 per cent. too high. Contrary to the statement in the text-books that ferrocyanides cannot be quantitatively oxidized with iodine, the athors show that satisfactory results can be obtained by using an excess of iodine lution and titrating back with thiosulphate. In presence of organic matter rtrates) the oxidation is not complete. Ferricyanides are reduced by boiling in rongly alkaline solution with ferrous sulphate, and in an aliquot portion of the ltrate, the ferrocyanide produced is titrated.

O. H.

Old and New Reactions of Ozone. C. Arnold and C. Mentzel. (*Berichte* 1902, xxxv., 1324-1330.)—Zinc iodide starch solution, potassium iodide starch solution, and guaiacum tincture are not characteristic reagents for ozone, since they also become blue on contact with chlorine, bromine, or nitric oxide.

Houzeau's test, too, which is based upon the blue coloration of red litmus-paper, dipped in potassium iodide solution, is quite unreliable, for the authors found that all the commercial test-papers examined by them became blue on contact with a dilute solution of iodine. They therefore made experiments to determine the value of other indicators as reagents, and found that only phenolphthalein, rosolic acid, and fluorescein were applicable.

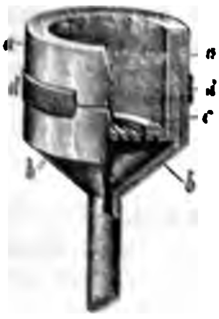
Paper saturated with a mixture of a 15 per cent. solution of potassium iodide and with a sufficient quantity of a 1 per cent. alcoholic solution of phenolphthalein to render the liquid opalescent, was coloured a fugitive red by ozone, whilst chlorine, bromine, or nitric oxide only gave blue or brown colorations. In like manner paper moistened with a mixture in equal parts of a 15 per cent. solution of potassium iodide, and a 1 per cent. alcoholic solution of rosolic acid became bright red on contact with ozone. The fluorescein test-paper was prepared by moistening black paper with a 1 per cent. solution of potassium iodide and a solution of fluorescein. It gave a green fluorescence in the presence of ozone.

Erlwein and Weyl's phenylenediamine test did not prove satisfactory in the authors' hands, though they found paper moistened with a mixture of paraphenylediamine and potassium acetate solutions to give characteristic reactions, yielding on contact with ozone a green colour changing to brown, and a blue or violet colour with chlorine, bromine, or nitric oxide.

Benzidene was found to be a very distinctive reagent. Test-paper prepared by moistening paper with a saturated alcoholic solution of that substance gave a brown coloration with ozone, whilst nitric oxide and bromine produced a blue coloration, and chlorine a blue colour changing to reddish-brown. Hydrogen peroxide was without action upon the benzidene test-paper, but a mixture of benzidene and copper sulphate solutions gave a blue precipitate with traces of hydrogen peroxide.

A still more sensitive and characteristic reagent for ozone was discovered in tetramethyl $p \cdot p'$-diamido-diphenylmethane. Test-paper soaked in an alcoholic solution of that compound became violet in the presence of ozone, deep blue with chlorine or bromine vapour, and faint yellow with nitric oxide, whilst it remained colourless on contact with hydrogen peroxide. C. A. M.

APPARATUS.



An Improved Büchner's Funnel. J. Katz. (*Chem. Zeit.*, 1902, xxvi., 356.)—As will be seen from the accompanying diagram, this apparatus is specially suitable for quantitative filtrations with the pump, as the space under the plate is accessible for cleaning purposes. *d* is a rubber ring sprung round the joint: thus the whole is made airtight without any danger of contaminating the solution with indiarubber. *c* is a porcelain ring which lies on the edge of the paper, and prevents it from being displaced. The funnel is made by F. Hugershoff of Leipzig. F. H. L.

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VOLUME XXVII.—No. 319.
OCTOBER, 1902.

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THE ANALYST.

OCTOBER, 1902.

OBITUARY NOTICE.

THE LATE SIR FREDERICK ABEL.

We regret to have to record the death of one of our honorary members, Sir Frederick Abel, which took place on September 6, at his residence in Whitehall Court. He was born in 1827, and commenced his chemical career in 1844 at the Royal Polytechnic Institution; this he left in the course of a few months, and became one of the first twenty-six students who entered the Royal College of Chemistry, which commenced its active career in 1845, under the direction of A. W. von Hofmann. Here he spent six years, for the greater part of the time acting as one of Hofmann's assistants, and left to succeed Faraday as Professor of Chemistry at the Royal Military Academy. In 1854 he was appointed Chemist to the War Office, which position he held for thirty-four years, during which time he devoted much attention to the study of explosives. Sir Frederick devised the first apparatus, known as the "open test" instrument, for determining the flash-point of petroleum, and this apparatus was legalized in the Petroleum Act of 1868. The indications of the instrument having been found unreliable, Abel brought out an improved apparatus, the "closed test" instrument, which was adopted as the basis of the British standard of 73° F. in 1879. He rendered valuable service as a member of the Council of the Inventions Exhibition of 1885. On the formation of the Imperial Institute two years afterwards, Sir Frederick was appointed secretary and general director, which duties he continued to perform until quite recently. He was elected a Fellow of the Royal Society in 1860, and was awarded a medal by that body in 1887. He served at various times as President of the British Association, the Chemical Society, the Institute of Chemistry, the Society of Chemical Industry, etc. The University of Cambridge, in which he filled the office of Rede Lecturer, conferred the honorary degree of D.Sc. on him in 1888, and Oxford that of D.C.L. in 1883. Finally, he was made C.B. in 1877, a Knight in 1883, K.C.B. in 1891, a Baronet in 1893, and G.C.V.O. in 1901.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

NOTE ON A SIMPLE APPARATUS FOR APPROXIMATELY ESTIMATING THE COLOURS OF WATERS.

By W. T. Burgess, F.I.C.

(*Read at the Meeting, March* 5, 1902.)

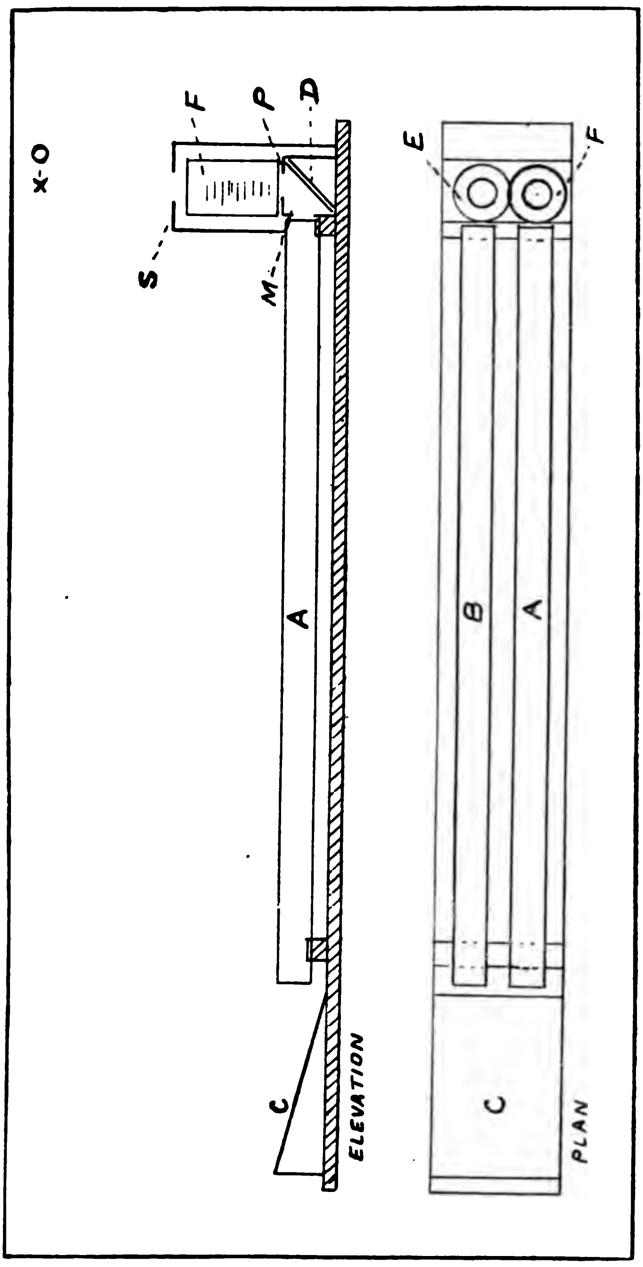
Several instruments capable of being used as colour meters for waters have been described, but have not come into general use, probably owing to the initial cost of the apparatus. I have found the inexpensive arrangement described below very convenient, and probably it may be of service to others, who, like myself, have occasion to make systematic examinations of water-supplies.

The water to be observed is contained in a horizontal 2-foot tube A (see figure) by the side of a similar tube B, containing distilled water; light, reflected from an inclined white card C, passes through both tubes, and a mirror D, fixed at an angle of 45°, reflects it upwards, so that the tints of the waters may be seen and compared by the observer's eye at O, about 10 inches above D. The ends of the 2-foot tubes rest against a mask M (having two circular holes 19 millimetres diameter), which screens off all light from the mirror except that passing through the tubes. Above the mirror D is a little platform P, provided with two circular openings, 19 millimetres diameter, and supporting two short cylindrical glasses E and F, 70 millimetres high and 40 millimetres diameter. A few c.c.'s of distilled water are placed in the cylinder F (through which the water under examination is observed), and a standard brown solution is run into the other cylinder E (above the distilled water 2-foot tube) until the colour of the water in A is matched. The depth of brown solution necessary is recorded in millimetres, read off either from a scale on the side of the cylinder E, or by immersing a small transparent glass or celluloid millimetre rule in the brown solution itself.

To facilitate the comparison, the mirror D is divided transversely, and the two halves slightly tilted and adjusted so that the images from both tubes may be observed simultaneously, without moving the eye, at O; and in making the final adjustment with the standard brown solution the cylinders are covered with a cardboard box screen S, provided with two viewing holes in the top.

The standard brown solution which I have found most convenient and generally applicable is the same as that now used in the colour meter originally described by Crookes, Odling, and Tidy (*Chem. News*, 1881), and consists of a solution containing 1 gramme of pure crystallized cobalt sulphate and 0·05 gramme of potassium bichromate in 1 litre. Occasionally the tints of waters are better matched by a somewhat browner solution containing 1·5 grammes of the cobalt sulphate and 0·05 gramme of potassium bichromate to the litre. The solutions keep well, but should be checked occasionally.

The 2-foot tubes are of stout glass, about 26 millimetres external diameter; on one end a disc of nearly colourless glass is cemented; round the other end a strip of thin celluloid (10 millimetres wide, and in length 10 millimetres less than the circumference of the tube) is tightly bound by wire, so that the strip projects about millimetre beyond the tube. To fill, the tube is held vertically, and completely illed with the water; a disc of thin glass placed on the open end is held in position



by the celluloid and surface tension, and after wiping the outside of the tube it may be placed on its side, or even inverted, without any risk of leaking. Possibly small tubes of similar construction will be found useful for polarimeters. All the glass discs for the long tubes, and also the discs for the bottoms of the glass cylinders E and F, should preferably be cut from the same thin sheet of colourless glass.

As in the case of all colour meters for water, turbidity, even if present in the slightest degree, introduces a disturbing element, and all water samples should, therefore, be examined in a large flask in a good light, and any opalescence noted.

Discussion.

The President said that, while comparison tests of the colour of water samples probably could not be utilized to any very great extent by the ordinary water analyst who had to deal with a large number of samples of different origin, such tests would be of considerable value to those who were dealing with one kind of water which did not alter very much in colour, and which might be taken as a standard or compared with a standard from time to time. It was important, however, to consider not merely the colour itself, but its probable cause. The cause might be an entirely harmless one—such, for instance, as peat—or, on the other hand, it might be due to sewage pollution. In isolated cases, therefore, too much stress must not be laid upon the colour itself without careful consideration as to its cause.

Mr. Richmond said that his own experience led him to believe that there was often a very distinct difference in colour between water polluted with animal matter and water coloured, for instance, by peat or decaying vegetable matter. One improvement which he thought might be worked out in the case of the apparatus that had been described was the use, instead of a standard solution, say, of potassium chromate and cobalt sulphate, of solutions of the three primary colours. The preparation of such solutions had been recently described in connection with a process of colour photography, and their use would afford a valuable means of estimating colour and expressing it in terms of the three primary colours. Accurate colour estimations would be found useful in other directions. For instance, in the determination of the end-points of titrations extremely accurate results could be obtained by means of a colour estimation. Of course, it was imperative that the solution should be quite clear. Sometimes, when turbidity occurred, colour effects were obtained in the case of solutions which had absolutely no colour. In order rapidly to remove turbidity in water by settling, it was necessary that the distance the particles had to travel should be as small as possible, and therefore it was an advantage to let the water stand in as thin a layer as possible; if the water were put into the tube and allowed to stand, the turbidity would settle down in a much shorter time than would be the case in a bottle.

Mr. Allen inquired whether the author had any experience of the use of coloured glasses such as were employed in Lovibond's tintometer. He would also like to hear what Mr. Richmond understood by the three primary colours—whether red, yellow, and blue, or red, green, and violet—and how he would propose that they should be obtained.

Mr. Richmond said that the primary colours he referred to were red, green, and violet, or blue-violet—*i.e.*, the three colours which, when they were made into screens and the light was passed through the screens separately and then combined, yielded a white or neutral gray light.

Mr. Allen inquired how the purity of the colours would be insured.

Mr. Richmond said that, as a matter of fact, the colours could be obtained absolutely pure if necessary, and colours which, though not absolutely pure, served satisfactorily as primary colours, could be obtained without much trouble. In a recent paper on the Lumière process of colour photography (abstracted in the *Journal of the Society of Chemical Industry*, 1902, page 275) were given the compositions of solutions by which the three primary colours could be prepared.

Dr. Rideal said that it was perfectly true that the estimation of the colour of water had most value when a comparison was being made between different samples from the same source; but, after all, that was frequently the kind of problem that had to be dealt with. Whether or not a water was good depended upon whether it remained constantly in the same condition, and whether it was similar to other waters in the same neighbourhood, so that variations of colour might be expected to throw some light upon the problem under investigation. In one case recently which he had had to deal with the colour of the water was certainly an important factor. In that case it had to be decided whether a peaty water, itself initially coloured, was contaminated with colliery water or with sewage or farmyard drainage. In addition to the colour imparted by the peat, the black colour from the coal, that due to urine from the men in the colliery, and from the farmyard contamination, all influenced the final colour of the waters. All of these colours were distinct, and although they all appeared to be more or less brown-yellow, the amount of each could be fairly accurately gauged by a Lovibond tintometer with varying tube-lengths. The brown-yellow colour of waters contaminated by any of these four different colours might be of approximately the same intensity in a tube 2 feet long, but if instead of using a tube of constant length one estimated the colours in tubes of different lengths, such as were used in polarimetric work, the fraction of absorption of the different colours would be different for each of the different colouring matters, and a very good differentiation could be arrived at by such means, even in cases where the colours were apparently the same when observed in a tube of fixed length.

Dr. Munro said that he had had occasion once to examine a spa water with a view to ascertaining whether the water was contaminated by the water of either of two small rivers which ran in front of the pump room only twenty or thirty yards from the well itself. One of these rivers was a limestone river, the bed of which was nearly pure carbonaceous limestone. The other was a boggy river deeply tinted with peat. It was perfectly easy, from a comparison of the colour alone of the three waters, to prove that the spa water was not contaminated from either of the other sources. It would not have been easy with merely a standard solution to go upon, because, although such a solution as had been mentioned in the paper would very fairly represent the colour of peaty water, it would not represent that of the limestone water, and still less that of the spa water. He had used in this case Lovibond's tintometer, by which with coloured glasses practically any tint or colour in water could be matched.

Mr. Burgess, in reply, said that it was, of course, impossible to form a proper opinion about a water merely from its colour, but he kept a record of the colour of all samples of water that he examined for reference in dealing with the same water subsequently. He had not had much experience with Lovibond's tintometer, but when he had tried it very carefully he had found it to suffer from one serious difficulty—namely, that in the case of waters containing very little colour the coloured discs themselves produced a dulness, and the light was considerably reduced in intensity. That was a difficulty which he, at any rate, had not been able to get over, and he had accordingly arranged the apparatus he had described, in which there was nothing to interfere with the brilliancy of the illumination. The standard solutions could be easily varied for different tints, and it would be easy to use tubes of different lengths, as suggested by Dr. Rideal.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

Refractive Indices of Salad Oils—Correction for Temperature. L. M. Tolman and L. S. Munson. (*Journ. Amer. Chem. Soc.*, xxiv., 754.)—The authors find that, using a Zeiss butyro-refractometer, the variation in the refractive index for 1° C. is practically constant at 0·000365 for ordinary oils, although the variation of the scale-reading of the instrument is not constant, since the scale is an empirical one. The most accurate way to correct scale-readings for temperature is to calculate the refractive index from the scale-reading found, correct it by means of the above factor, and then calculate back to the scale-reading. For variations of a few degrees only the formula—

$$R = R' - X(T - T')$$

may be used, where R = reading corrected at T, R′ = reading at T′, T = desired temperature, and T′ = temperature of actual reading, and X = change in scale division caused by a change in temperature of 1° C. For scale-readings of 40° to 50°, X equals 0·55; from 60° to 70°, 0·58; and from 70° to 80°, 0·62.

The oils examined were: olive, poppy-seed, maize, sunflower, rape, white mustard, black mustard, lard, pea-nut, cotton-seed, and sesame. A. G. L.

Composition of Kissi Pepper. A. Barillé. (*Journ. Pharm. Chim.*, 1902, xvi., 106-116.)—The pepper known as *Piper Famechoni-Heckel*, or Kissi pepper, is obtained from a new species of *piper* growing in French Guinea. The fruit resembles miniature bunches of grapes, and when pulverized yields a reddish-brown powder with a characteristic aromatic odour and a somewhat acid taste. It has been used for some time past by the Soudanese troops in place of ordinary pepper. According to the results of the author's analysis, it has the following composition:

	Per Cent.
Water	14·60
Ash, soluble in water	3·61
Ash, insoluble in water	0·94
Volatile oil	4·47
Piperine	3·70
Starch	38·00
Cellulose	10·01
Glucose	5·21
Saccharose	1·66
Proteids	10·24
Gums, colouring matter, and soluble nitrogenous substances	5·27
Resins and fixed oil	3·95
Alcoholic extract	19·25
Ethereal extract	16·07
Total nitrogen	1·82

C. A. M.

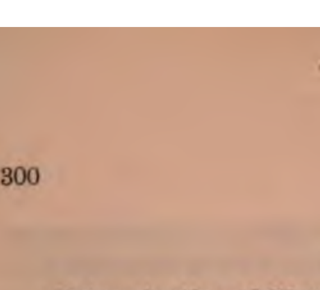
On the Determination of Aconitine in Certain Preparations of Aconite. H. Ecalle. (*Journ. Pharm. Chim.*, 1902, xvi., 18-23.)—In the case of aconite preparations made with glycerin and alcohol it is only possible to extract a very small proportion of the aconitine by treatment with ether. The author has therefore made experiments as to the possibility of effecting a complete precipitation of the alkaloid by means of silico-tungstic acid in the presence of a slight excess of nitric acid. He finds that the aconitine silico-tungstate is soluble to some extent in glycerin, and that it is necessary to use a large excess of silico-tungstic acid and nitric acid to prevent this. The amount of aconitine is found by multiplying the weight of the dried and ignited precipitate by the factor 0·793. C. A. M.

TOXICOLOGICAL ANALYSIS.

Notes on the Detection of Mercury in Cases of Poisoning. D. Vitali. (*Boll. chim. farm.*, 1902, xlii., 149; through *Chem. Zeit. Rep.*, 1902, 173.)—Mercuric sulphide, precipitated by sulphuretted hydrogen, is not wholly unattacked by nitric acid; if it is boiled for some time with equal quantities of nitric acid and water, it is partly oxidized and converted into nitrate, and this nitrate combines with the excess of sulphide to form a white double compound, which looks much like lead sulphate. This compound is identical with that already described by Rose, which was produced by treating a solution of mercuric nitrate with an insufficient quantity of sulphuretted hydrogen. If, then, the precipitate given by sulphuretted hydrogen yields a white powder with nitric acid, it must be investigated like the black sulphide of mercury. Vitali states that several undoubted cases of mercury-poisoning have occurred in which the metal has been missed on analysis, and he suggests that the above reaction is the cause of the error. F. H. L.

ORGANIC ANALYSIS.

Determination of Glycerol. S. Zeisel and R. Fanto. (*Zeits. landw. Versuchsw. Oesterr.*, 1902, v., 729; through *Chem. Zeit. Rep.*, 1902, 173.)—The author's process depends on the conversion of the glycerol into isopropyl iodide by means of boiling aqueous hydriodic acid, and the determination of the former as silver iodide. The flask *a* holds about 40 c.c., and its side tube is put in connection with a carbon dioxide apparatus. Into it is charged such a weight or volume of the substance to be analysed that the amount of silver iodide finally recovered does not exceed 0·4 gramme, together with 15 c.c. of a solution of hydriodic acid, having a specific gravity of 1·9 if the liquid is aqueous; otherwise of 1·7. The flask is surrounded with a glycerin bath, and made to boil gently. The condenser *b* is coupled to the Ehmann's warming apparatus *g*, and is kept full of water at a temperature between 50° and 70° C. The little flask *c* stands in a water-bath at the same temperature, and is one-third full of water containing some red phosphorus, or of a solution of potassium arsenite; *e* and *f* are provided with marks indicating 45 and 5 c.c. respectively, and they are filled to those marks with silver solution. The carbon dioxide current is passed at a speed of 3 bubbles per second. At the end of the distillation the contents of the receivers are rinsed out into a beaker, diluted to about 450 c.c., mixed with 10 or 15 c.c. of dilute nitric acid, heated on the water-bath, cooled, and the silver iodide is estimated either gravimetrically or volumetrically.



F. H. L.

Effect of Bleaching on the Constants of Beeswax. R. Berg. (*Chem. Zeit.* 1902, xxvi., 605.)—All processes of bleaching increase the acid number of beeswax, the natural method least, treatment with chromic acid most of all. Ordinary bleaching and permanganate leave the ester number unchanged, or slightly raise it; chromic acid always lowers it. The increase of the acid value is invariably so large as to lower the ratio; and without a qualitative analysis the bleached wax might be thought to be adulterated with stearin. The saponification value of bleached wax is also always a trifle higher than that of yellow wax. The influence of bleaching upon the iodine value is peculiar; contrary to expectation, treatment with chromic acid usually raises it; but Italian waxes exhibit a lower iodine absorption after any method of bleaching. The Buchner number is lowered a little by natural bleaching or by permanganate; chromic acid raises it, sometimes considerably. The Buchner number of Italian wax, however, falls during bleaching. Bleaching by the ordinary process or by permanganate does not affect the rotatory power of the wax, or has a tendency to increase it; but treatment with chromic acid, or any method of bleaching Italian wax, reduces the optical activity. Chromic acid frequently raises the melting-point notably; other processes lower it a trifle. "Kothwachs," a by-product in working up pure beeswax, which is really a mixture of bleached and unbleached wax with dust and dirt, requires such drastic treatment to purify it again that its constants show irregular variations before and after bleaching. F. H. L.

Boiled Hübl Iodine Solution. M. Kitt. (*Chem. Zeit.*, 1902, xxvi., 554.)—The author gives further details respecting the stability of the boiled Hübl solution which he has already recommended (ANALYST, 1902, vol. xxvii., 22). A quantity of solution was prepared, and was found to contain 0·76756 gramme of free iodine per 25 c.c.; next day, after boiling under the inverted condenser for five hours, its titre was 0·27743 gramme—a loss of 63·9 per cent. After keeping one year it still tested 0·24876 gramme, so that during storage it only lost another 3·7 per cent. of its original strength. A sample of linseed oil examined with ordinary Hübl solution gave an iodine value of 165 (mean); with year-old boiled solution, using 50 c.c. per 0·1 to 0·2 gramme of oil, the figures were 163·2 and 164·5. F. H. L.

Behaviour of Araban towards Fehling's Solution. E. Salkowski. (*Zeits. physiol. Chem.*, 1902, xxxv., 240; through *Chem. Zeit. Rep.*, 1902, 174.)—The author has previously stated that, whereas xylan is precipitated from its alkaline solution by Fehling's solution, araban is not thrown down. Experiments on gum arabic, however, have shown that in certain circumstances araban is also capable of precipitation. An excess of Fehling's solution hinders the precipitation of araban, since the potassium-copper compound of the latter is soluble in the reagent, although but slowly. The difference in behaviour of alkaline solutions of araban and xylan towards Fehling's solution is extremely small; but if only those substances are present, the character of the precipitate enables them to be distinguished. From a mixed alkaline solution of araban and xylan alkaline copper solution throws down both bodies, but the copper compounds may be separated by their different solubilities in cold water. The araban, which passes into solution, retains a little xylan; but the xylan may be freed from araban by repeated washing. F. H. L.

Examination of Paper - making Materials by the Pentosan Method. E. Kröber and P. Rimbach, communicated by B. B. Tollens. (*Zeits. f. angew. Chem.*, xv., 508.)—When the material contains, in addition to pure cellulose (linen or cotton rags), only one other material, such as wood-meal, or sulphite cellulose, the determination of the pentosans enables one to estimate the quantities of each present. Pure cellulose yields about 1 per cent. pentosan; soda cellulose, 6 per cent.; sulphite cellulose, 7 per cent.; wood-meal, 12 per cent. By the use of these figures a simple calculation gives the quantities of the constituents, provided these are qualitatively known. Newspaper material generally consists of wood-meal and sulphite cellulose. A. M.

Determination of Tannin. E. Crouzel. (*Repert. Pharm.*, 1902, [3], xiv., 248; through *Chem. Zeit. Rep.*, 1902, 174.)—The tannin is brought into solution and mixed with an excess of analgesin (dimethylphenylpyrazolone), together with twice as much of sodium bicarbonate. The opaque liquid gradually becomes clear and green, a precipitate falling. This is collected on a weighed filter, dried at 100° C., and weighed. The amount of analgesin equivalent to a certain quantity of tannin is ascertained by adding the former as long as a precipitate falls. The process might be made volumetric, but the end-point of the reaction is difficult to see. F. H. L.

The Estimation of Indigotin on Fabrics. A. Binz and F. Rung. (*Zeits. angew. Chem.*, 1902, xxiii., 557.)—A further examination has been made of the method of extracting the indigotin by means of glacial acetic acid (see ANALYST, vol. xxiv. 48). Experiments performed with pure indigo showed that in the process of extraction a portion of the dye stuff is destroyed, but this loss does not amount to more than a few tenths of a per cent. calculated on the weight of fabric, and is to a certain extent compensated by the fact that some of the fabric itself is extracted by the acetic acid. The method therefore yields satisfactory comparative figures. A. M.

Estimation of Mineral and Organic Impurities in Hard (Varnish) Resins. J. Hertkorn. (*Chem. Zeit.*, 1902, xxvi., 602.)—Owing to the lack of proper methods for the examination of the hard resins which are employed in varnish manufacture, it is customary to run samples through a number of sieves of specified gauges, preferring the materials according as they contain a higher percentage of large lumps, with chips or broken particles, and only a small quantity of dust. Since, however, varnish resins are costly, the dust and chips have to be used, and it is desirable that they shall contain as little foreign matter as possible. The impurities in broken copal and copal dust may range between 0·5 and 50 per cent.; but more than 5 per cent. of organic matter is unusual, 0·3 to 2·5 per cent. being the average, with 1 to 5 per cent. of mineral substances. Most objectionable to the varnish-maker among the organic impurities are humus-like bodies, fragments of wood, leaves, etc., since these are decomposed at a temperature of 300° C., and spoil the colour of the varnish. The author suggests the following method of extracting the resins from the accompanying noxious ingredients: A solvent is prepared by mixing together from 20 to 25 parts of amyl acetate, 40 to 50 parts of amyl alcohol, and 25 to 40 parts of ethyl alcohol of more than 96 per cent. strength. In a large light beaker 5 to 10 grammes of the sample, powdered as finely as possible, and if necessary first run through a cloth, are weighed out, and stirred carefully with 25 to 50 c.c. of the solvent, avoiding the production of lumps. The beaker is next placed in water at 70° or 80° C., still stirring till the liquid boils, and allowed to remain under cover for thirty or sixty minutes. When the residue appears sandy and no longer resinous it is set aside in a warm place to settle till clear, and the solution is run off into another vessel. The insoluble matter is then treated as before until the liquid ceases to leave a residue on evaporation, passing the washings through a paper tared at 105° (filtration being necessary as the less concentrated liquors do not settle clear). Finally, the precipitate on the filter is washed twice with ethyl ether, dried to constant weight at 105° C., and weighed. Care must be taken that the paper does not become dry during the process. If the residue is still cohesive the whole extraction must be repeated. The residue may be used for a determination of ash, or the latter may be determined on a separate portion of the sample. F. H. L.

Experiments with the Kjeldahl Method of Nitrogen Estimation. H. D. Law. (*Journ. Soc. Chem. Ind.*, 1902, xxi., 847.)—The nitrogen was determined in a sample of leather, heating with sulphuric acid for different times in the presence of different oxygen carriers. When no oxygen carrier was used, but only potassium sulphate and acid, the solution did not always become clear even after one and a half hours' heating, and unless it did become so the result was low. In another series of estimations 2 grammes of copper sulphate were introduced in each case; after one hour the solutions were clear, and then constant results were obtained. With 1 gramme of potassium permanganate correct results were also obtained after heating half an hour only, but with both these carriers much precipitate is formed on adding alkali, and bumping consequently occurs. To avoid this difficulty the author used potassium persulphate instead. The leather was first digested for a quarter of an hour with sulphuric acid alone; the mixture was allowed to cool slightly, then 10 grammes of the persulphate were added, and the liquid was shaken up well and the heating continued. The reaction proceeded smoothly. In half an hour the solution was clear, and gave the correct result. A. M.

Determination of Hydrocyanic Acid. A. Archetti. (*Chem. Zeit.*, 1902, xxvi., 555.)—The solution containing the cyanide is shaken in an Erlenmeyer flask with a known excess of mercurous chloride and the mixture is filtered. The insoluble matter, consisting of metallic mercury and the excess of calomel, is then treated on the paper with a liquid composed of 2 volumes of 1·40 nitric acid and 1 volume of water, which attacks the mercury only; the residue of mercurous chloride is washed with water, dried, and weighed. The difference gives the amount of cyanide according to the relationship $Hg_2Cl_2 = 2HCN$. F. H. L.

INORGANIC ANALYSIS.

Notes on the Estimation of Copper by Potassium Permanganate. H. A. Guess. (*Journ. Amer. Chem. Soc.*, xxiv., 708.)—The following modification of the permanganate method for copper, which does away with the use of asbestos as a filtering medium, and consequently requires no filter-pump, is stated to give results at least as good as the iodide or cyanide methods, especially in the case of small percentages of copper. From 1 to 5 grammes of the material (ores, tailings, etc.) are digested with aqua regia to which a few drops of sulphuric acid have been added, the excess of acid is boiled off, and the solution diluted and filtered. The filtrate is neutralized with ammonia, rendered just acid with hydrochloric acid, and reduced by adding an excess of sodium sulphite solution, after which a slight excess of potassium or ammonium thiocyanate is added, the whole heated to boiling, and then, as soon as the precipitate has settled, filtered through an extracted 11-centimetres filter-paper, the precipitate being well washed with hot water. The funnel containing the precipitate is removed to another vessel and filled twice with a boiling 10 per cent. sodium hydrate solution, which decomposes the cuprous thiocyanate. After thoroughly washing the insoluble cuprous hydroxide left on the filter, the filtrate and washings, which contain the sodium thiocyanate, are rendered strongly acid with sulphuric acid, and titrated whilst still warm with potassium permanganate. This latter should be standardized against pure copper treated as above, as, owing to the slight solubility of the cuprous thiocyanate, the value calculated from the iron value of the permanganate is a little too low. The small error caused by the action of the caustic soda on the filter-paper may be determined and allowed for by treating an empty filter in the same way and titrating the filtrate. A. G. L.

Rough Estimation of Tin in Tin-dye Baths. O. Geisel. (*Chem. Zeit.*, 1902, xxvi., 553.)—From solutions of stannic chloride, not containing too much free hydrochloric acid, and of a strength equivalent to between 0·075 and 0·1 gramme of $SnCl_4$ per 10 c.c., cold sodium hydroxide throws down all the metal before the acid is neutralized. The author therefore proposes to mix such a solution with a few drops of a 1 per cent. solution of malachite green, and to titrate the liquid with decinormal soda till the colour changes from the greenish golden-yellow of the lake to the original bluish-green tint of the dye. The operation may be carried out in a large beaker standing on a white ground, or, better, by the usual colorimetric method of comparison. Gravimetric experiments show that the calculation of the amount of tin present should be made according to the equation:

$$7NaOH + 2SnCl_4 = Sn(OH)_4 + SnCl(OH)_3 + 7NaCl.$$

F. H. L.

Volumetric Estimation of Small Quantities of Iron. F. Seiler and A. Verda. (*Chem. Zeit.*, 1902, xxvi., 804.)—When a solution of ferric thiocyanate is treated with potassium ferrocyanide, Prussian blue is formed, the red colour of the first compound, which quickly becomes brown, changing sharply to green, which afterwards turns to blue, when the whole of the thiocyanate is decomposed. The reaction proceeds according to the equation:

$$3[Fe(CN)_6K_4] + 2Fe_2(CNS)_6 = [Fe(CN)_6]_3Fe_4 + 12KSCN,$$

so that 4·92 grammes of ferrocyanide are equivalent to 1 gramme of iron. A potassium ferrocyanide solution is therefore prepared of such a strength that 1 c.c. is equal to 0·001 gramme of iron. The substance to be analysed is brought into solution in the form of ferric chloride, and a suitable quantity of the liquid is mixed with an excess of ammonium thiocyanate. The whole is diluted with water till a bright red colour results, and titrated with the ferrocyanide till a green colour appears. The authors give two examples of their method as applied to a medicinal powder containing iron, and a sample of red wine, the figures showing it to be exact.

F. H. L.

The Determination of Sulphur in Pig-iron by Eschka's Method. John V. R. Stehman. (*Journ. Amer. Chem. Soc.*, xxiv., 644.)—In this method 3 grammes of the powdered sample are mixed with 2·5 grammes of a mixture of 2 parts magnesia and 1 part sodium carbonate in a platinum crucible; the whole is covered with a layer of 0·5 gramme of the same mixture, and heated to a red heat for one hour. A gasoline burner is preferable for heating, since, if ordinary coal-gas is used, a shield must be employed to keep the products of combustion away from the mouth of the crucible. After allowing the mass to cool, it is treated with hot water and 10 c.c. bromine, the whole boiled for fifteen minutes, filtered, and, after the addition of 1·5 c.c. concentrated hydrochloric acid, the bromine is expelled from the filtrate by boiling, and the sulphur is precipitated with 10 c.c. of a 10 per cent. barium chloride solution.

The sample used for this method must be at least fine enough to pass through a 60-mesh sieve. Using a steel mortar, pestle, and hammer, this only requires five minutes for a 20-gramme sample. Results obtained in this way are practically identical with those obtained by three of the older methods. A. G. L.

On the Quantitative Separation of Zirconium from Iron. A. Gutbier and G. Hüller. (*Zeits. Anorg. Chem.*, xxxii., 92.)—The solution of iron and zirconium is precipitated hot with a small excess of ammonia and the precipitate filtered off; the filtrate is evaporated to dryness on the water-bath, the residue dissolved in a little acid, a small excess of ammonia added, and any resulting precipitate of zirconium hydroxide filtered off and added to the main precipitate, which is then dried and ignited at a red heat. The increase in weight of the crucible gives the weight of $Fe_2O_3 + ZrO_2$. The oxides are then very finely powdered in an agate mortar and heated to constant weight in a platinum boat in a current of hydrogen at a red heat. By this means the iron is completely reduced to the metallic state, the zirconium dioxide not being affected. From the loss in weight the quantity of ferric oxide, and hence that of zirconium dioxide, present in the original mixture of oxides may be calculated. A. G. L.

The Volumetric Determination of Molybdenum in Molybdenum Steel and Ferro-Molybdenum. James Brakes. (*Journ. Soc. Chem. Ind.*, 1902, xxi., 833.)—This is a modification of Kopp's method (see ANALYST, xxvii., 202). Weigh out 5 grammes, dissolve in 20 c.c. nitric acid and 20 c.c. hydrochloric acid, evaporate to dryness, add 20 c.c. of dilute sulphuric acid (1 : 1), and evaporate until white fumes appear. Cool, and then proceed as in Kopp's method (*loc. cit.*). In calculating the result, subtract the blank, then multiply the remainder by the iron equivalent of the permanganate solution and by the factor 0·605, as is indicated by the equation:

$$5Mo_{12}O_{19} + 17K_2Mn_2O_8 = 60MoO_3 + 17K_2O + 34MnO.$$

The results agree with those obtained by Whitfield's gravimetric and Kopp's methods. Tungsten should be removed before evaporation with sulphuric acid. Chromium does not affect the results even if present to the extent of 8 per cent. A. M.

On the Quantitative Separation of Zinc and Cobalt. Arthur Rosenheim and Ernst Huldschinsky. (*Zeits. Anorg. Chem.*, xxxii., 84.)—The authors have tried various methods for the separation of zinc and cobalt, and obtained the best results by precipitating the zinc as sulphide from an acetic acid solution of the double cyanides. In this method a small excess of potassium cyanide and a considerable amount of ammonium chloride are added to the neutral solution of the metals, which is then boiled for a short time to form the complex potassium cobalticyanide, and after the addition of acetic acid zinc is precipitated by means of hydrogen sulphide. Fairly good results were obtained by precipitating the zinc as sulphide from dilute solutions of the two metals to which acetic acid only had been added. The separation of cobalt as potassium cobaltinitrite, on the other hand, gave unsatisfactory results, even when both the solutions of the metals and of the potassium nitrite were acidified with acetic acid before mixing, as about 1 per cent. of the zinc appears to be precipitated with the cobalt. A. G. L.

The Cloudiness which occurs on Filtering Zinc Sulphide. O. Mühlhaeusser. (*Zeits. angew. Chem.*, 1902, xxiii., 731.)—Zinc may be entirely precipitated as sulphide, if mineral acid be first neutralized with ammonia and the solution be acidified with acetic acid before passing sulphuretted hydrogen. If ammonium acetate be also added the danger of loss of zinc is further reduced. When the sulphide is filtered off the filtrate is always cloudy, but this cloudiness is wholly due to sulphur. Known quantities of zinc were precipitated as described, and the precipitates were filtered off under various conditions, washed with a solution of acetic acid and sulphuretted hydrogen, and dissolved in hydrochloric acid. The solutions were then titrated with ferrocyanide, and in every case the correct amount of zinc was found. A. M.

The Marsh-Berzelius Arsenic Deposit. W. Ackroyd. (*Journ. Soc. Chem. Ind.*, 1902, xxi., 900.)—The colour of the arsenic deposits in the tube is liable to vary; in the case of organic materials they are practically always brown, but when inorganic substances are tested the deposits are often wholly or partly blue. This difference the author ascribes to variations in the rate at which the arsenic is evolved. When the velocity of the hydrogen in the tube is small, the blue deposit, if formed, is nearer the generator than the brown, but if the velocity be great the relative positions are reversed. The author considers that the brown colour is the normal one. In order to be able to measure and regulate the flow of gas, he arranges a platinum wire with its end 3 millimetres from the jet. The diameter of the flame should not exceed 3 millimetres, else there is danger of obtaining a blue deposit. A. M.

Analysis of Crude Sulphur. F. P. Carpenter. (*Journ. Soc. Chem. Ind.*, 1902, xxi., 832.)—To remove calcium sulphate treat with dilute hydrochloric acid, boil for about ten minutes, filter on a Gooch filter, and dry. The sulphur can be determined in this residue either by extracting with carbon disulphide, or by burning and ascertaining the loss. The latter method gives results which are slightly too high, because some organic matter is removed at the same time. The calcium sulphate, when present, prevents the sulphur being completely dissolved by carbon disulphide, but when it has been removed this difficulty disappears. A. M.

A Volumetric Method for the Estimation of Sulphuric Acid in Soluble Sulphates. Yasujuro Nikaldo. (*Journ. Amer. Chem. Soc.*, xxiv., 74.)—The method depends on the precipitation of the sulphate in dilute alcoholic solution by means of standard lead nitrate solution, potassium iodide being used as indicator, the yellow colour of lead iodide becoming permanent only when all the sulphate has been precipitated. For $\frac{N}{10}$ solutions it is best to work in 60 to 70 per cent. alcoholic solutions, and for $\frac{N}{2}$ solutions in alcohol of 50 to 60 per cent. strength. The quantity of potassium iodide added should be about 0·2 gramme for 25 c.c. liquid. Solutions should be cold, not too dilute, and should not contain free nitric or hydrochloric acids, sodium acetate, or large quantities of chlorides. In the presence of heavy metals the addition of acetic acid is desirable to prevent precipitation of basic compounds. A. G. L.

Determination of Sulphuric Acid in Soils. C. B. Williams. (*Journ. Amer. Chem. Soc.*, xxiv., 658.)—In the method prescribed by the Association of Official Agricultural Chemists sulphuric acid is estimated by directly adding barium chloride to the acidulated soil solution. The author, however, points out the necessity of first removing the iron and aluminium by means of ammonia, since their salts, usually present in considerable amounts, exercise a solvent action on the barium sulphate, which is important, considering the small quantities of sulphuric acid usually present in soils. In thirty-five experiments in which the official method was compared with his modified one the results obtained with the latter were, on an average, more than 35 per cent. higher than those given by the former. A. G. L.

The Use of Persulphates in Analysis. H. D. Dakin. (*Journ. Soc. Chem. Ind.*, 1902, xxi., 848.)

Estimation of Manganese.—Ammonium persulphate was substituted for bromine in the usual method of precipitation in ammoniacal solution. Working with pure solutions containing known amounts of manganese very satisfactory results were obtained. When fixed salts are present, considerable quantities are carried down with the precipitated oxides of manganese. As in the bromine method, this difficulty must be guarded against. As the precipitate varies much in its degree of oxidation, it is not possible to estimate the manganese volumetrically from its oxidizing power.

Oxidation of Chromium Salts.—Chromic salts are readily converted into alkaline chromates by boiling with a large excess of caustic alkali and adding potassium persulphate until oxidation is complete. The solution may then be acidified with nitric acid and precipitated with mercurous nitrate, and the precipitate filtered, washed, and ignited in the usual way. Ammonium persulphate cannot be used in this case, as it does not oxidize the chromium completely. A. M.

On the Analysis of Gases by Combustion. Walther Hempel. (*Zeit. Anorg. Chem.*, xxxi., 445.)—From some results obtained by him, the author concludes that mixtures of, *e.g.*, methane with large quantities of nitrogen are not completely burned when mixed with air and passed repeatedly through a red-hot capillary tube containing platinum, or over a red-hot platinum spiral, unless the gas mixture is actually capable of explosion. This condition may be usually fulfilled by adding sufficient oxygen, but in mixtures very poor in combustible gases the addition of hydrogen as well as oxygen becomes necessary. In any case, the analysis of gas mixtures by explosion appears to be safest. A. G. L.

The Estimation of Dissolved Oxygen in Water. William Naylor. (*Chem. News.*, lxxxv., 259.)—The estimations are carried out by Gerland's method (*Journ. Soc. Chem. Ind.*, xv., 15; xviii., 340), which depends on the reduction of indigo by sodium hyposulphite, as follows: About 50 to 100 c.c. tap water are placed in a four-necked Woulff's bottle, the air in which is then replaced by coal-gas which has been passed over heated copper turnings. The top of the hyposulphite burette, which is fitted into one of the stoppers of the Woulff's bottle, as well as the reservoir of hyposulphite solution, are also filled with this deoxidized coal-gas. The coal-gas escapes from the bottle by means of a tube in connection with a small water wash-bottle. When the bottle is full of coal-gas 1 or 2 c.c. of the indigo solution, which should contain 0·0005 gramme available oxygen per c.c., are run into the bottle by means of a tap-funnel, the end of which dips below the surface of the water, and exactly reduced by the hyposulphite. If the solution remains a pale straw-colour, the gas in the bottle is free from oxygen, and the hyposulphite solution is next standardized against the indigo. One hundred c.c. of the sample are then run in, and the quantity of hyposulphite necessary to destroy the blue colour produced noted. Nearly two and a half times this amount of hyposulphite solution is then run into the bottle, followed by 250 c.c. of the sample; the faint blue colour produced is removed by a few drops more of hyposulphite, and the total for the 250 c.c. ascertained.

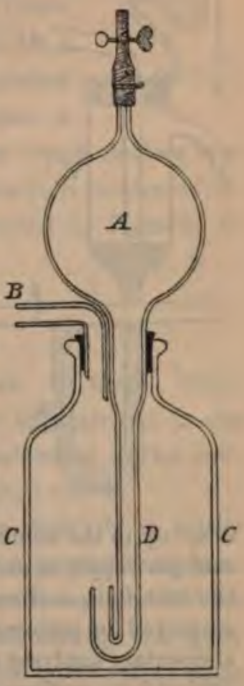
The pure indigo, before being used, must be washed with water, hydrochloric acid, and alcohol, and then treated with ammonium persulphate in acid solution. By this method a hundred samples could be examined in one day without washing out the Woulff's bottle, the solution being removed from time to time through a syphon tube. A. G. L.

The Stability of Potassium Tetroxalate and Sodium Oxalate as Standard Substances for Volumetric Analysis. — Dupré and A. von Kupffer. (*Zeits. für angew. Chem.*, 1902, xv., 352.)—These substances have been recommended for use to standardize both oxydimetric and acid solutions, the salt being weighed out and converted into carbonate by ignition. The tetroxalate may also be used directly to standardize alkaline solutions. Five different samples of potassium tetroxalate were tested at intervals. Of these three were bought and two were prepared from potassium oxalate and oxalic acid only. One of the specially prepared samples only gave constant results, the others all gradually losing strength as determined by standard permanganate solution. The strength as determined by titration with standard acid was, however, constant in each case, though the numbers for different samples are not identical.

In the case of sodium oxalate a purchased sample lost only 0·2 per cent. in fifty-nine days as determined by permanganate. A sample prepared from pure oxalic acid and soda gave a constant result, 99·36 per cent. The one satisfactory sample of tetroxalate gave the same figure. The permanganate was standardized by means of electrolytically deposited iron. The authors consider that sodium oxalate may be used with advantage, but not the tetroxalate. A. M.

APPARATUS.

A Modification of Hempel's Apparatus for Gas-Analysis. Theodore William Richards. (*Proc. Amer. Acad.*, xxxvii., 201; through *Zeits. Anorg. Chem.*, xxix., 359.)—The following simple pieces of apparatus have been designed for use in teaching gas-analysis, instead of the more expensive and cumbersome forms of Hempel. The pipette is replaced by a bulb mounted by means of a long tube in the bottle containing the absorbent reagent, as shown in the figure. To prevent the entry of air from below, the tube is narrowed at its lower end, and bent upwards. For the introduction of solid reagents the tube *D* is made wider and fitted with a stopper pierced by one hole, through which a small bent tube is inserted. For fuming sulphuric acid, the bulb should be ground into the neck of the bottle, which must also be fitted with another neck carrying a-ground in side-tube. If 50 c.c. of gas are used for the analysis, the volume of the bulb *A* should be about 75 c.c., and that of the bottle *C* 150 c.c.



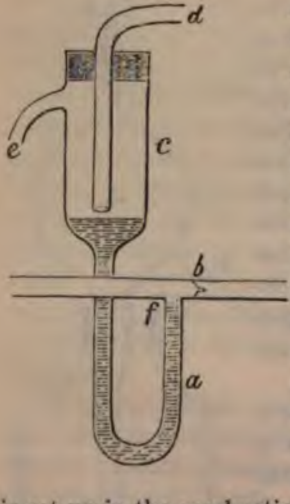
Hempel's burette may be replaced by an ordinary burette, either inverted, with the stop-cock on top, or else in its normal position, in which case the upper end is fitted with a rubber stopper, through which a capillary tube is inserted, care being taken that the stopper always occupies the same position. The ungraduated upper part of the burette must, of course, be carefully calibrated. The small size of the burette permits it to be readily surrounded by a water-jacket, which entirely obviates the errors arising from a change of temperature.

The manipulation of this apparatus is practically the same as that of Hempel.

In analysing about 50 c.c. of mixtures of air and carbon dioxide with it, the average error of seven determinations was only 0·04 c.c. (0·00 to 0·10 c.c.). A. G. L.

A Chemical Method for obtaining Reduced Pressures. Francis G. Benedict and Charlotte R. Manning. (*Amer. Chem. Journ.*, xxvii., 340.)—To obtain high degrees of exhaustion the authors have successfully employed the following method: Into the upper part of a Hempel desiccator of a total capacity of 3·5 litres 150 c.c. of concentrated sulphuric acid are poured. The dishes, etc., containing the material to be dried are next introduced, and finally 10 c.c. of pure anhydrous ether are delivered from a pipette upon the bottom of the desiccator. The cover is immediately put on, and the desiccator connected with a water pump until a pressure of 40 to 60 millimetres is indicated by a small manometer placed inside the desiccator. The stop-cock of the desiccator is then closed, when after a few minutes a vacuum of from 4 to 1 millimetres is obtained. This degree of exhaustion is reached in eight to twelve minutes after putting on the cover of the desiccator. The sulphuric acid may be used several times without losing its power of absorbing the ether vapour. A. G. L.

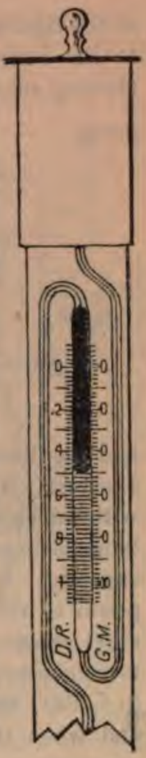
Governor for Use during Ultimate Analysis. I. Deiglmayr. (*Chem. Zeit.*, 1902, xxvi., 520.)—The object of the device shown in the accompanying illustration is to bring the ordinary process of combustion under automatic control, thus avoiding the necessity for watching the gas current, and for constantly manipulating the burner cocks. It is essentially a thermostat, placed, in the case of carbon and hydrogen determinations, between the water and the carbon dioxide tube. The main gas-supply pipe of the furnace is divided into two portions, gas entering at either end; that which heats the part where the oxidant alone is put passes direct from the service, but the gas which feeds the burners under the substance being analysed first enters the thermostat through *d*, and then escapes through *e*. The horizontal tube in the diagram is the connection between the sulphuric acid vessel and the potash bulbs, and it is reduced at the point *b* to a capillary of suitable bore. If, then, combustion is proceeding so quickly that there is danger of absorption not being complete, the current of carbon dioxide is too rapid to pass *b*, and pressure is set up in the combustion tube sufficient to force the mercury in *a* up into *c* till the coal-gas supply is choked. Thus the gas flames become smaller, the temperature of the tube falls, and combustion is rendered slower. An identical arrangement can be employed in nitrogen estimation. The original article records a large number of elementary analyses carried out with the aid of the apparatus described, these analyses having been performed without any attention being given to the furnace after it was once started, in some instances the operation proceeding unwatched for a considerable



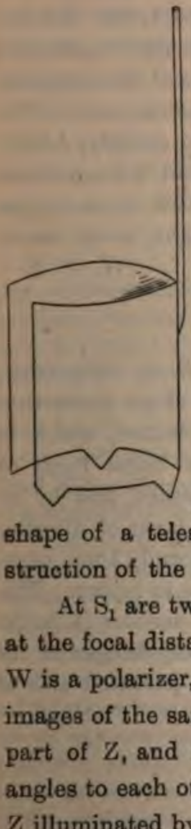
period of time. When the device is adopted, combustions should be conducted without a current of oxygen or air until the process is nearly complete. F. H. L.

Beckmann's Thermometer with Kühn's Additional Scale. (*Chem. Zeit.*, 1902, xxvi., 337.)—By means of the additional scale attached to the upper portion of the thermometer (see figure) it is easy to determine the necessary amount of mercury required for any given interval in the scale attached to the capillary tube. The auxiliary scale is so graduated in relation to this latter scale that, for example, when it records 50° the thread of mercury reaches to about the middle of the capillary $\frac{1}{100}$° scale. The necessary amount of mercury to be taken from the reservoir is thus directly known.

C. A. M.

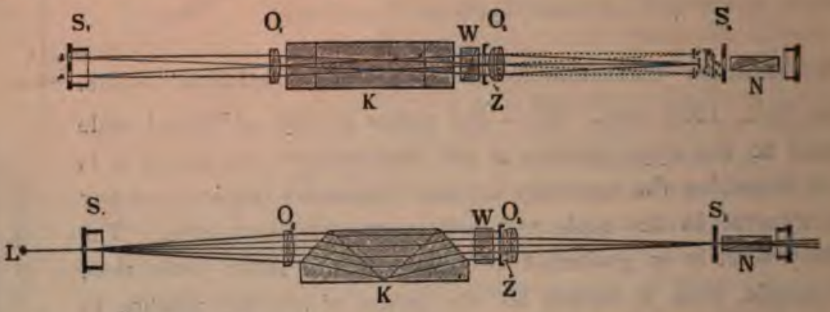


An Improved Platinum Electrode. (*Chem. Zeit.*, 1902, xxvi., 356.)—Krause recommends a platinum electrode made as shown by the annexed sketch. The imperfect cylindrical form makes it convenient for insertion and removal from the liquid, renders it a useful stirrer, and causes the deposited substance to be thrown down as a uniform layer. The electrode is constructed by Heraeus of Hanau. F. H. L.

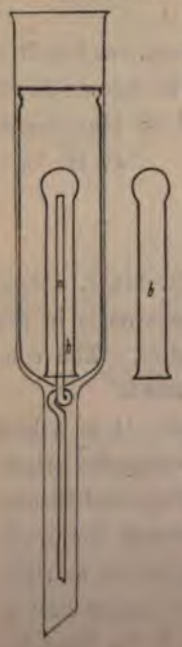


A New Form of Pyrometer (Wanner's). R. Hase. (*Zeit. für. angew. Chem.*, 1902, xv., 715-717.)—The apparatus is in the shape of a telescope, 30 centimetres long, and acts as a photometer. The construction of the optical part of the instrument is shown in Figs. 1 and 2.

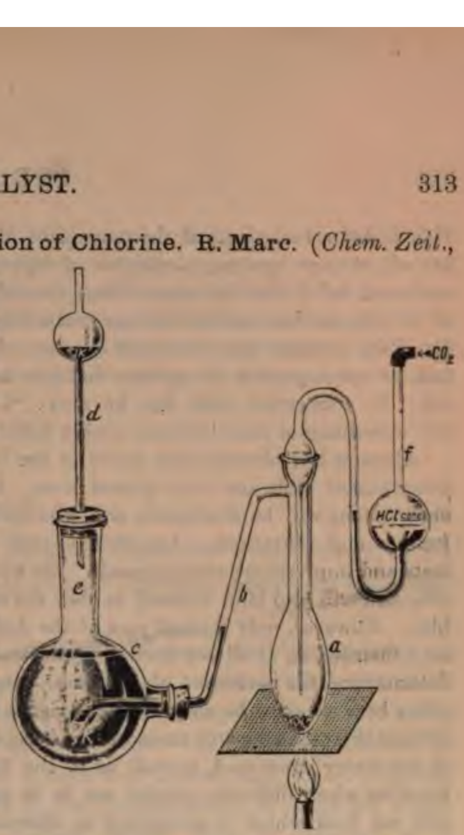
At S_1 are two slits, *a* and *b*, placed vertically one above the other; O_1 is a lens at the focal distance from S_1, for making the rays parallel. K is a rectangular prism. W is a polarizer, and Z is a double prism. The lens O_2 collects the rays and throws images of the same directly on the slit S_2. The rays from *a* pass through the upper part of Z, and those from *b* through the lower half. Both are polarized at right angles to each other, and the eye, observing the images at S_2, sees the upper half of Z illuminated by *a* and the lower half by *b*. By rotating the analyser N, the intensity of the light on the two halves may be equalized. A 6-volt incandescent lamp is fixed to the instrument, and lights one half of Z for comparison. The light from the glowing substance of which the temperature is to be determined enters the other slit, and the illumination is made equal in intensity to that of the lamp by turning the analyser. The reading of the scale of the instrument is then taken and compared with tables on which temperatures corresponding with the readings are given. The lamp is lighted by a 0·8 ampère current from an accumulator of 10 ampère-hour capacity. When the accumulator has run down to a certain extent, the lighting power of the lamp is altered, but may be corrected by comparison with a standard amyl acetate lamp. This operation is the work of a minute. Some of the temperatures observed by the author are interesting. Thus, molten iron showed from 1,372° to 1,384°; the same, when run from the furnace and commencing to solidify, 1,230°, and when the surface was solid, 1,012°; glass-smelting ovens had temperatures between 1,530° and 1,630°. A zirconium plate heated in an oxygen blast had a temperature of 2,090°; and an arc-light, using retort-carbon, 3,610°. W. P. S.



A Modified Soxhlet. G. Schüle. (*Chem. Zeit.*, 1902, xxvi., 653.)—The annexed sketch shows the construction of an extraction apparatus specially designed for the analysis of butter, which is claimed to exhibit several advantages over the usual form of Soxhlet. The inverted tube is detachable, as indicated, and by its central position, surrounding the syphon, allows a layer of filtering material to be packed into the extractor more uniformly than heretofore. Being inside, too, these tubes are less liable to injury. The constriction at the upper end of the main vessel enables it to be laid safely on its side in the drying oven without any danger of its liquefied contents running out. The apparatus is patented and is made by F. Mollenkopf, of Stuttgart. F. H. L.



Apparatus for the Volumetric Estimation of Chlorine. R. Marc. (*Chem. Zeit.*, 1902, xxvi., 556.)—This simple apparatus, which obviates some of the inconveniences attached to Bunsen's apparatus, generally used for this purpose, is designed for the examination of such substances as are attacked but not completely decomposed by cold hydrochloric acid, such as yield a solution which is not suited for titration, or such as are conveniently analysed for other constituents in their original solution after the chlorine has been driven off. The joint between the hydrochloric acid bulb and the boiling flask is ground; that between the receiver and the check bulb might also be ground if desired, but most of the chlorine is absorbed in the main vessel, and the residue is so highly diluted with carbon dioxide that this refinement is not necessary. The method of using the apparatus is obvious.



F. H. L.

A GOVERNMENT SCIENTIFIC BOARDING-HOUSE.

(*Reprinted from the* "*Times*" *of September* 29, 1902.)

[FROM AN AMERICAN CORRESPONDENT.]

One of the advantages which the United States has had, especially since it became fairly populated, has been that it is apparently an inexhaustible field for experiments. The latest of these is, perhaps, the most novel of all. It is neither more nor less than the opening, by the authority of an Act of Congress, of a boarding-house which is to be conducted upon purely scientific principles. It has for its declared purpose the testing of the effect of various preservatives, colouring substances, and other food admixtures upon persons supposed to be in good health. A good deal of ridicule has already been wasted upon this proposal, but Dr. H. W. Wiley, chief of the Division of Chemistry in the Department of Agriculture, under whose auspices this experiment is to be carried on, has recently given an outline of the work which he intends.

According to this announcement, board will be provided for twelve individuals. These are to be chosen upon scientific principles, and for this purpose resort will first be had to the various bureaus of the Agricultural Department, and, after them, to the college students resident in Washington. It is intended to maintain the tables for months, and perhaps for years. It is Dr Wiley's purpose to do something towards settling the controversy over the healthfulness of various articles almost universally used in everyday diet. It is well known that medical authorities, whose business in life it seems to be to differ each from the other, have condemned one by one every article consumed by man. This controversy has raged with especial bitterness whenever, proceeding beyond the domain of purely natural products, an attempt is made to enlist the services of the chemical laboratory for the preservation of food. It is therefore one of the purposes of the experiment to find out the relative harmfulness of various articles.

This is defined as a part of the movement towards pure food legislation. For instance, borax has all at once become a question of diplomacy. The German Government, acting upon its professed belief that American meats treated with it are harmful, in spite of the contrary view of its own medical authorities, has practically prohibited the importation of meats thus treated. Dr. Wiley assumes that the small quantity of boric acid used in curing meat is not harmful. In fact, he has expressed the opinion that it is decidedly less hurtful than an equivalent quantity of salt. In connection with this, he says: "I believe this, but I do not know it. The object of this experimental table is to test such a belief in the most practical way."

Allusion has already been made to the fact that the boarders to be accommodated at this Government table are to be picked men. It is recognised that they must be persons whose observations will be intelligent, and who have the power to express with some accuracy their feelings and sensations. Accordingly, each boarder will keep a diary, and record all sorts of facts and impressions about himself. He will be put upon his honour to eat nothing anywhere else, and will also bind himself to obey the scriptural injunction, and to eat what is set before him. However, only a small part of the desired information will come from the records of the men themselves, as all the devices understood by the medical profession will be resorted to for determining the perfection of digestion. Among the rules which will be observed is one that every boarder shall be weighed upon rising from bed in the morning. Three times a day the clinical thermometer will measure and record his temperature. A careful account will be kept of the water consumed, as well as of the food. The boarders will not have the advantage of knowing when different articles are to be tried upon them. For at least half the time they will eat food which is recognised as thoroughly pure. This is to be known as a relaxation diet. The object of this will be not only to prevent any real injury to the system, but also to tell how far into a period of normal conditions the effect of former harmful ones may persist. At each meal, also, some men will be eating the treated, or doctored, food, and some pure food, divided as the physician may direct. The quantities of adulterants employed are not to be perceptible at any time to the senses, although a doubt is expressed as to the ability to carry out this rule when it comes to colouring matters.

The preservatives for these experimental tables will be prepared and applied to the food by experts, and the quantity employed in each particular instance will be measured with the greatest accuracy. The detailed effects towards which the inquiry will be directed will concern various organs of the body and known constitutional tendencies to certain diseases. For instance, salicylic acid will be subjected to all the demands which, in the commercial movement of food, it is ever likely to make on the physical systems of American consumers. It is expected that the tabulated results will throw light upon the degrees of danger, and also define and fix the limits of safety, if any, in the use of this particular acid. It is intended that the same process shall be applied all the way through the list of many inventions which man has sought out in articles of diet. A curious feature of the experiment, as explained by Dr. Wiley, is that an attempt will be made to keep the boarders the same weight during their entire stay at the table, as a fluctuation in this respect might add a confusing element to the result. When it is discovered from the daily weighing that the subject is gaining a little, his ration will be so adjusted in its fat-producing elements that this tendency will be corrected, and the food at all times will be so generally wholesome and appetizing that no one in ordinary health need expect to lose weight.

This brief outline of an experiment which is being subjected to serious consideration and to trial, seems, as already explained, to be a part of a movement for the suppression of adulteration. At the present time this is a function belonging to the State Legislatures, with the result that a great deal of confusion has already been produced. For the last fifteen years a Bill has been pending before Congress for the enactment in a Federal law of the various provisions now incorporated in the State laws, rejecting those which have been shown to be impracticable.

Thus, standards of strength in vinegar, cream, and all sorts of articles, are now different in different States. This legislation has, it is explained, become very annoying, especially to the manufacturers of honest goods, so that there is more of a willingness than has heretofore been manifested that the Federal Government should enact a law to cover all these questions.

If the experiment which has been explained is not subjected to too much ridicule, there will probably result from it some reports which, whatever their value, will at least be novel and interesting.

REVIEWS.

WATER-SUPPLY: CONSIDERED PRINCIPALLY FROM A SANITARY STANDPOINT. By WM. P. MASON. New York: Wiley and Sons; London: Chapman and Hall. Price $4.

This work, which is the third edition of Professor Mason's well-known book, is a welcome addition to the library, as, although the chapters relating to the chemical and bacteriological examination of water are omitted in consequence of their being published separately, the work deals on broad lines with the general question of water-supply under varying conditions. After a review of the ancient water-supplies and the Roman aqueducts, the author proceeds to the consideration of drinking water and disease, more especially in relation to typhoid fever. This is followed by a review of the various processes for the artificial purification of water and various sources of supply, such as rain, river, and stream water, stored, ground, and deep-seated water, a consideration of the daily quantity required per head of the population, and the action of water upon metals. Whilst it cannot be said that there is any considerable quantity of novel information contained in the text, which forms most interesting reading, it must be confessed that the author has gathered together a great deal of scattered information upon the general question, with the result that a most useful work is produced, which is thoroughly worth the careful study of those seeking information on the subject. The statistics referring to American experience with which the book abounds constitutes a most important feature. A carefully compiled index completes a work which we are glad to have on our bookshelf.

W. J. D.

A TEXT-BOOK OF INORGANIC CHEMISTRY. By DR. A. F. HOLLMAN. New York: Wiley and Sons; London: Chapman and Hall. Price 10s. 6d.

This is, in many respects, a most excellent and interesting work. It is well got up, and is commendably free from printers' errors; is up to date as to facts, and adequately deals with modern views and theories. The theoretical portions are, indeed, the feature of the work. Theories are very fully and clearly discussed, and are far more frequently used in explanation of reactions described than is usual in manuals of chemistry. We could, indeed, have wished that these portions had been treated consecutively, and had not been scattered over the whole work, sometimes without any apparent reason for the place in which they are found, but in this the author follows the example of most text-books.

The descriptive portions are not quite so good; there are some strange omissions, and here and there we come upon what we can only characterize as somewhat careless writing or reasoning which, in an author who, as a rule, has evidently taken great pains to be strictly logical and correct, is rather surprising.

The production of oxygen from barium peroxide is not mentioned, and, indeed, no process for the production of oxygen on a large scale is given. Six methods for the preparation of hydrogen are briefly described, but its preparation by the passage of steam over red-hot iron is not among them, though, in another part of the book, allusion is made to the reaction involved.

On page 3 we are told, without any qualification, that physics deals with temporary, chemistry with lasting, changes of matter; whereas, in many, if not in all, cases, the temporary or lasting character of the change depends on the temporary or lasting character of the condition which has brought about the change. Ice melts at 0° C., and remains water as long as the temperature remains between 0° C. and 100° C.; mercury, when heated for some time to near its boiling-point in contact with oxygen, becomes converted into mercuric oxide which, when the temperature is raised, breaks up into mercury and oxygen; yet the latter is a chemical, the former a physical change. Many similar cases will occur to the reader.

On page 10 we are told that the fact that a glowing splinter, when introduced into an atmosphere of oxygen, begins at once to burn brightly, is ordinarily used as a characteristic test for oxygen. If this is so it is a mistake, as should have been pointed out by the author; the facts are correctly stated under nitrous oxide. On page 24 we find it stated that when an electric current is passed through water to which a little sulphuric acid has been added, two volumes of hydrogen are evolved for every 1 volume of oxygen; as a matter of fact there is always a slight deficiency of oxygen.

We have noted some more instances of the same nature, but the above will be sufficient to illustrate our meaning; they are, after all, but minor blemishes easily corrected in a new edition, and we can heartily recommend this text-book to teachers and advanced students.

A. D.

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VOLUME XXVII.—No. 320.
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THE ANALYST.

NOVEMBER, 1902.

OBITUARY NOTICE.

DR. JOHN HALL GLADSTONE.

WITH great regret we have to chronicle the death of one of our honorary members, Dr. J. H. Gladstone, which occurred suddenly on October 9. He was born in 1827, and was consequently in his seventy-sixth year. He studied chemistry first at University College under Graham, afterwards at Giessen under Liebig. He graduated as Ph.D. in 1848, and was elected F.R.S. in 1853.

Dr. Gladstone was the first president of the Physical Society on its formation in 1874, and was president of the Chemical Society from 1877 to 1879. His first paper was published in 1845, and from that time onwards he devoted his constant attention to scientific research, maintaining for many years a laboratory, with assistants, at his own expense. He engaged himself largely with chemistry and optics, and the points of contact between these two sciences. Thus, he was a pioneer in a line of work that has been much developed in more recent times, and which has led to important results. Amongst his investigations may be mentioned that of the solar spectrum, and his determination of the optical constants of a very large number of bodies; to him we owe much of our knowledge on the refractive indices of the essential oils. For his optical and electrical researches, together with much other work, he was awarded the Davy Medal of the Royal Society a few years since. So lately as the present year two papers—one written by himself, the other in conjunction with Mr. W. Hibbert—were read at the meeting of the British Association held in Belfast.

Dr. Gladstone was for some time Lecturer on Chemistry at St. Thomas's Hospital, and afterwards Fullerian Professor of Chemistry at the Royal Institution. He was the author of several books, amongst these "The Biography of Michael Faraday" and "The Chemistry of Secondary Batteries."

Dr. Gladstone was also engaged for many years in various philanthropic movements. From 1873 to 1894 he was a member of the London School Board, acting as vice-chairman for three years.

His death terminates a long career of solid, useful work in many directions, always carried on with unostentatious modesty and urbanity. His genial and kindly presence will be greatly missed at the many scientific meetings where he was a well-known and constant attendant.

THE PROPOSED STANDARDIZATION OF METHODS OF CHEMICAL ANALYSIS.*

By Bertram Blount, F.I.C.

There has been evident of late years a tendency to apply the principle of standardization to matters and in directions differing considerably from those in which the advisability of erecting and enforcing a standard is generally conceded.

The commonest examples of standardization — namely, that of weights, of measures, and of money—have come into existence and are maintained by their manifest necessity. Various mechanical standards are so convenient as to have become practically necessities; for instance, standard gauges for wire and sheet have been used very freely, and standard screw threads have also been found desirable.

But, passing from these simpler cases where standardization is universally conceded to be convenient to a consideration of the advisability of extending this principle to more complex matters, we enter into a region of polemic; the advantages of an artificial uniformity are less clear, and the disadvantages become more easily discernible and are ultimately prominent. As an instance of this may be mentioned the proposal to standardize sections of structural metals, sizes of test-pieces for mechanical tests, and methods of applying such tests.

If the difficulty of accepting the principle of standardization is considerable in the matter of mechanical testing, it becomes still greater in the case of chemical analysis.

In mechanical testing, it is recognised that the values obtained for strength in tension and compression, for elongation of a ductile test-piece, for resistance to shock and the like, are appreciably influenced by the conditions of the test, and, in consequence, there is something to be said in favour of standard methods of applying the tests. But there is no such justification for the adoption of standard methods in chemical analysis where the object in view is the determination in a given material of a definite substance; in such a case all methods which are chemically sound must give the same results.

But notwithstanding these obvious facts, a desire has arisen in some quarters for the formulation of standard methods of chemical analysis, and for their gradual imposition on the community of chemists; steps have been taken to secure this end, and it is necessary to decide whether this movement is laudable or not, lest judgment should go by default.

The wish to establish standard methods of analysis is no new thing. A serious proposition in this sense was put before the Society of Chemical Industry by Professor Lunge in the year 1884. Discussions followed in which leading chemists, many of whom are still with us, took part, and in the face of an evident conflict of opinion it became necessary to appoint a committee to consider the whole question. I have not been able to find that any report by this committee was published, and from the situation to-day it is fairly certain that no definite decision was arrived at.

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* Abridged from a paper read before the British Association at their meeting in Belfast, 1902.

About the same time, now nearly twenty years ago, the Society of Public Analysts occupied themselves with a similar subject. The methods of water analysis were discussed, and recommendations for a standard process were formulated. A like course was adopted for the analysis of milk, and some years later for the detection of butter in margarine by the Reichert-Wollny process.

It will be observed that in all these cases the substances to be examined are natural products of indeterminate composition, and that the methods for their examination are of the nature of empirical tests rather than of analysis proper. Such tests are often of the highest importance and utility for practical purposes, and from their nature are best performed under standard conditions, but they must not be confused with actual chemical analysis, which is under no such obligation to rule.

At the meeting of the British Association held in Bath in 1888 a committee was appointed "to consider the best methods of establishing international standards for the analysis of iron and steel." The initiation of this investigation appears to have been due to Professor J. W. Langley, who undertook the preparation of the necessary samples. Other national committees were invited to join in the work, and after some years certain of those committees reported. At an early stage of the proceedings the British committee defined the object of the inquiry as the provision and preservation of specimens of iron and steel of a composition most carefully ascertained. These were intended to serve as standards whereby the accuracy of any given process or any given worker might be checked. It will be observed that there is here no word of a standard method of analysis; the members of the committee were to be free to use the method they thought proper, and the results alone were to be the standard. But the members of the American committee seem to have held a different object in view; they applied themselves to the examination of methods, and especially of those for the determination of carbon. It may be conjectured that their objective was the provision of a standard method, but this aim was not attained, and no subsequent attempt in the domain of steel analysis has met with greater success. It is interesting to note that whilst no standard method of steel analysis has been arrived at, the improvement in existing processes and the device of new methods have continued without pause or set-back, and that this branch of analytical work has been brought to a high state of efficiency.

But the idea, although not realized, was by no means dead. C. B. Dudley, of the Pennsylvania R.R., in 1893 discussed the causes of discrepancy between the results of different chemists, and advocated the use of standard methods for avoiding these discrepancies. Quite recently (in 1901) a committee of the New York section of the Society of Chemical Industry approached the same subject in the same spirit, and has reported in favour of a standard method of analysis for cement and cement materials. I had the honour in May last of reading a paper before this body, in which I ventured to criticise the method in detail, and to dissent from the acceptance of a standard method.

These criticisms and objections were very frankly and fairly received and discussed, and it may be hoped that the discussion will induce some of those who were perhaps over ready to except standardization on its superficial merits to consider its real and weighty disadvantages, and to confine their activity to those branches of analytical work in which some form of standardization is admittedly useful.

The introduction of standard methods for the analysis of coal, sugar, fertilizers, and feeding-stuffs and for water analysis has also been advocated in recent years, chiefly in the United States, and some of the proposed methods have been officially adopted by various associations.

These instances will suffice to show that there is an increasing tendency to propose standardization of analytical methods, irrespective of the nature of the material to be analysed, and it remains to consider how this tendency has arisen and what effect it is likely to have if it continues to grow without opposition.

In the first place, it will be seen that there are in this matter two schools of thought sharply differentiated. On the one hand, we have chemists so impressed with the necessity of avoiding discrepancies that they are anxious to set up standard methods, stated in such detail that a faithful observance of the prescriptions by two workers cannot fail to secure identical results. And there are others less confident of the success of this system. This conflict of opinion arises in great measure from confusion of thought. There are many operations performed by the chemist which are from their nature arbitrary. To take an extreme case, it is evidently impossible to carry out the Maumené test for oils or to determine the flashing-point of petroleum, and to obtain concordant results without having recourse to a fixed procedure. In the examination of potable waters such arbitrary methods are in wide and general employment, and yield much useful information. Feeding-stuffs and manures afford a similar case. "Citrate-soluble phosphoric acid" is a meaningless term unless the conditions of its extraction are laid down. The fractional distillation of crude benzol and the return of its computed contents of benzene is another instance.

But this sort of codification, though useful and often necessary, has nothing to do with analysis. The object of the analyst is to determine with the best precision in his power the constituents of the substance which he is analysing. Sometimes he cannot do this, and is forced to have recourse to the determination of the properties of the substance; that is to say, he is compelled to apply to the material under examination various arbitrary tests. Standardization for these tests is legitimate enough; standardization of analysis implies that analysis is an arbitrary procedure not based on ultimate chemical facts. This view is too grotesque for discussion.

To turn to the much-debated question of discrepancies in analytical results, which is at the root of the whole matter.

In trade it is necessary frequently to appeal to the analyst, and if the results given by one analyst differ from those of another, examining what is believed to be the same sample, much delay and expense may be incurred. The trader is usually unaware of the great difficulty which exists in procuring identical samples, and he is almost certainly ignorant of the slow and laborious character of all analytical processes having claim to be exact. From the former cause he is disposed to ascribe all differences to errors of the analyst, and from the latter he is inclined to urge greater rapidity of work than is compatible with accuracy. Sometimes from bad sampling or from hurried or unskilful work discordant or erroneous results may arise. Then the trader seeks a remedy, and believes that it may be found in a standard method of analysis. It need hardly be urged before this assembly that such a conclusion is fallacious. The same imperfection in sampling, the same hurried and unskilful work will lead to the same erroneous results; the only difference will be that each Chemikant—for he cannot be called a Chemiker—will be entitled to say that he had followed the prescription as faithfully as he was able.

It is not likely that chemists who practise as independent consultants will submit to dictation, even from a committee of their peers, as to the methods they are to employ. A member of this branch of the profession, asked to determine in some material a definite constituent, is either competent to choose a suitable method, and to execute it accurately, or he is not. If he is, a standard method is superfluous; if he is not, his early retirement is desirable, and will probably occur from the operation of natural causes. But there are some consultants who are more or less identified with particular branches of the chemical trade; they are constantly called upon to carry out analyses or assays for the buyers and sellers of ores, metals, minerals, salts and the like, and usually the results obtained by the buyer's and the seller's chemist are compared, and may be found to disagree. Now, here is a plausible case for the use of a standard method. It is argued that if the two chemists concerned were to use the same process the likelihood of their disagreement would be diminished. There is even a limited truth in this; the likelihood of disagreement would be diminished to some extent because both operators would be exposed to identical chances of error, instead of different chances. But the object of analysis is not to obtain a false concord by repeating a process, errors and all, with Chinese fidelity; it is to arrive at the truth as closely as knowledge and skill will allow, and one of the recognised methods of doing this is to vary the conditions of experiment. How misleading are mere concordances is sufficiently shown by some of the earlier determinations of atomic weight vitiated by a systematic error, exposed at once on radically changing the method. If the atomic weight of nitrogen had depended on the determination of the density of the residuum obtained by eudiometric analysis, a considerable error would have been admitted in a fundamental figure for a period of many years.

The need of the buyer's and seller's chemist for identical results will best be met by insuring that they are sufficiently well trained to refuse to employ a faulty method; their choice among several sound methods is obviously a matter of indifference. It is only when the assay is from its nature arbitrary, and is, in fact, a conventional test, that an identity of method is necessary.

A great part of the clamour for standardization arises from the discrepancies which often occur between the results of different works' chemists, and it appears that the "standard" methods which have from time to time been proposed in the United States have the works' chemist as their particular objective. These discrepancies are highly inconvenient, not only immediately to those concerned, but ultimately as tending to discredit the value of analytical control of manufacturing processes. When last in America I took some pains to ascertain the opinion of chemists on this point, and found that those who advocated the adoption of standard methods, when confronted with the grave objections which can be urged against them, took refuge in the necessities of the works' chemist, maintaining that only by the provision for him of standard methods could uniformity and accuracy be attained.

It cannot be denied that this view is defensible, but it is defensible on one ground alone, and that hardly likely to commend itself to the works' chemist. His friends, in their anxiety to help him, infer that he is incompetent, and needs to be guarded and guided like a schoolboy. Now, as a matter of fact, although often overburdened with work and most inadequately paid, the works' chemist has done much to advance analytical chemistry, originating some methods and improving many others. It is neither just nor courteous to assert that he, above all others, is in need of direction and supervision from without, and that he is willing to barter his right of individual judgment, according to his training and experience, for a bundle of prescriptions.

It is, however, unfortunately true, that while there is nothing in the nature of things why the works' chemist should not be as highly trained and as capable of individual decision as his consulting colleague, yet it remains a fact that some works' chemists are not fully competent, and, conscious of their deficiency, might even welcome the promulgation of a standard method by which a given analytical process shall be carried out. Such cases are freely advanced by the advocates for standardization, and doubtless afford some show of argument in favour of the movement. But surely it is better and sounder to direct effort, not towards the provision of rules for half-trained chemists, but towards insuring that the chemists are fully trained. And here one is met by the money question. At present the average manufacturer is so little alive to the absolute necessity of obtaining proper scientific and technical aid in the conduct of his business that he will offer to the chemist a salary hardly in excess of the wages of a skilled mechanic. Is it to be wondered at that the man who is willing to accept such remuneration should not be fully trained, that his knowledge should be empirical, and that he should need the prop of a "standard" method scientifically on a par with a cookery recipe? If it is the desire of those who advocate the standardization of chemical methods to promote the multiplication of analytical machines turning out results of artificial uniformity, no better course could be adopted than the prescription in detail of processes which shall relieve their user from the troublesome process of thinking. I do not like to suppose that this is the object deliberately desired, but it is well at least to recognise that it is likely to be attained by the successful establishment of standard analytical methods.

There are other results to be expected of wider and more general importance. Let us suppose that standard methods had been prepared and published with the consent and under the direction of some body of chemists of sufficient standing to make their pronouncements authoritative. The position of all chemists who ventured to disagree with the findings of this central body would become very difficult. In all cases of dispute, in all legal proceedings, these dissentients would be placed at a disadvantage; on them would be thrown the onus of proving, usually before a non-technical tribunal, that the method they preferred was as good as that of the central committee. Now, it is as certain as anything not actually accomplished can be, that the standard method at its best would be a compromise between the different processes in use by the various specialists in that particular branch of work, and would have the usual quality of a compromise—namely, the preference of expediency for principle. Consequently, no independent worker basing his process on what he conceived to be the soundest principles could avoid a conflict with a method which from its origin is a hybrid product. If the dissentient were not able to convince the lay tribunal that his process was reliable, a grave injustice might be done to him; if, on the other hand, he did so succeed, the standard process would be discredited. A whole vista of trouble and dispute can be foreseen. The endeavour here made to trace to its logical outcome the effect of standardization is sufficient to show that such a scheme, even if it could be regarded as desirable, would lead to such complications as to render it impracticable.

But there are still further drawbacks of a kind yet more serious. If a standard process is adopted by a central authority, it will be regarded by many as final, and all incentive to original inquiry in that direction will be removed. Periodical revision by a standing committee will probably be carried out, but naturally on the lines of the existing process, and with the view of improving it in detail. The deviser of a method radically different from that which is official will be regarded as a sort of chemical heretic, and will receive the treatment usually accorded to the heterodox. Now, analytical research, although not the highest form of chemical investigation, is of great value both as a school of training and as a means of perfecting the chief instrument of the chemist, whose work, unless constantly controlled by its aid, would be little better than a maze of ingenious speculation. Anything calculated to discourage the criticism of existing analytical methods and the device of new ones is, therefore, much to be deprecated.

There is too good a case against the standardization of methods of chemical analysis to make elaboration of argument necessary or wealth of instance and illustration desirable. The plain facts constitute so crushing an indictment, and appeal so strongly to every thinking chemist who is jealous of the dignity of his chosen profession, that their statement without ornament or rhetoric must suffice for conviction. But as there is, nevertheless, a respectable body of opinion in the contrary sense, it may not be superfluous to register a formal pronouncement that standardization is undesirable except in cases where arbitrary methods of examination cannot be avoided.

To turn to the construction of a positive policy. The spirit which is dissatisfied with the present status of the analyst, that does not hesitate to indicate the causes of errors and discrepancies, and is eager to incur serious and voluntary labour in order to diminish the frequency of their occurrence, is so entirely admirable that if directed towards a better end than the construction of a cramping and ineffective code it could not fail to animate a larger and more liberal project with its own zeal. There are many branches of analysis (as distinct from arbitrary methods of chemical examination) in which there still exists some doubt whether certain of the recognised methods are actually as reliable as the users of them maintain. It would be outside the scope of this paper to put forward a list of these discussable methods; every practising chemist can provide illustrations from his daily work, and the specialists in any well-defined branch of analytical work would, if they agreed in nothing else, be thoroughly in accord with the idea that revision of many accepted processes, after proper experimental inquiry, might be usefully undertaken. To realize this idea it seems to me that there might be formed standing committees of chemists interested in particular branches of analytical work; that those committees should meet as often as is practicable, in order that their members might confer; that each should draw up a programme of work to be carried out by selected members of the committee, the programme to be directed to the examination of existing methods of analysis, to the elimination of sources of error which might be discovered, and to the device of new and improved processes; that the results of the deliberation of these committees should be published periodically, in order that chemists at large might be informed of the progress made and warned against the errors discovered. All these inquiries would be of vast value to professional chemists, who would be in a position to judge how far the methods indicated might usefully be received and adopted.

It will be seen that the programme suggested differs radically from that to which I have ventured to take exception. The latter involves the pronouncement of a defined method for the determination of a given substance; the former is directed to the discovery of latent errors, and to the provision of processes free from these. In the one case a set of rules is imposed from without; in the other, advance and improvement will take place from within, unfettered by canon and unhampered by rule. I think it not too much to say that the whole spirit of science, which is instinct with the love of freedom and the defiance of mere authority, will be found favourable to the plan which I have put forward, and will unconditionally condemn any attempt to standardize methods of chemical analysis.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

The Composition of Sheep's Milk. **Trillat and Forestier.** (*Ann. de Chim. anal.*, 1902, vii., 321-323.)—The authors have made analyses of a large number of samples of French sheep's milk taken during the months of February, March, and April. The following results are given as typical:

	Per Cent.	Per Cent.	Per Cent.	Per Cent.
Total solids dried at 100° C. ...	20·03	19·58	18·90	18·56
Fat	7·40	7·42	6·98	7·18
Lactose	5·32	5·35	5·53	5·26
Casein	6·18	5·87	5·54	5·12
Ash	1·021	0·394	0·961	1·018
Calcium oxide	0·247	0·256	0·250	0·238
Acidity	3·7	3·0	2·66	2·8

These figures are considerably higher than the mean of the results obtained by Chevalier and Henri, Doyère, Gorup-Besanez, and other authors, viz.: Total solids, 12·4; fat, 4·2; lactose, 4; casein, 3·7; and ash 0·7 per cent. C. A. M.

The Determination of Lecithin in Milk. F. Bordas and S. de Raczkowski. (*Ann. de Chim. anal.*, 1902, vii., 331-333.)—The authors consider that the most rational method is to determine the glycerophosphoric acid in the milk in the following manner: One hundred c.c. of the milk are shaken with a mixture of alcohol (95 per cent.), 100 c.c.; water, 100 c.c.; and acetic acid, 10 drops. The coagulum thus obtained is filtered off and extracted with three successive portions of 50 c.c. of hot absolute alcohol. The united extracts are evaporated, and the residue dried on the water-bath and taken up with a small quantity of a mixture of equal parts of alcohol and ether. The filtered solution is evaporated to expel the ether, the residue saponified with potassium or barium hydroxide, and the soap solution decomposed with very dilute nitric acid. After separation of the fatty acids the filtrate is evaporated to dryness, and the residue mixed with 10 c.c. of concentrated nitric acid, and oxidized on the hot-water bath by the gradual addition of pinches of powdered potassium permanganate. The precipitated manganese hydroxide is then dissolved by the addition of a few drops of sodium nitrite solution (1 : 10), the nitrous vapours expelled by boiling, and the phosphoric acid precipitated with ammonium molybdate, and finally determined in the form of magnesium pyrophosphate. The weight of pyrophosphate thus obtained, multiplied by the factor 1·5495, gives the amount of glycerophosphoric acid in 100 c.c. of the milk. C. A. M.

The Colouring Matter and Sugar of Apricots. A. Desmoulière. (*Ann. de Chim. anal.*, 1902, vii., 323, 324.)—Truchon and Martin-Claude (ANALYST, xxvi., 102) examined the expressed juice of various fruits, including apricots, but did not find dextrose or any colouring matter soluble in acid or ammoniacal amyl alcohol in any of them. In repeating part of this work the author examined four samples of apricots of different origin, and found dextrose in each. In the two cases in which quantitative determinations were made the following results were obtained:

Sugar.	Fruit not completely Ripe. Per Cent.	Ripe Fruit. Per Cent.
Saccharose	3·131	3·814
Invert sugar	2·383	2·299
Dextrose	0·771	0·353

The apricots, pounded in a mortar with water, yielded a liquid from which a yellow colouring matter could be extracted by either acid or alkaline amyl alcohol. On evaporating the extract a yellow residue was left, which, on the addition of a drop of sulphuric acid, became indigo blue, changing to brownish-violet. This reaction is identical with that given by carotin, the colouring matter of the carrot. The colouring matter of the apricot can be distinguished from coal-tar colours by the fact that it does not dye silk or wool. C. A. M.

On the Influence of Cane-Sugar and Dextrin in the Bees' Food upon the Composition of the Honey. Von Raumer. (*Zeit. anal. Chem.*, 1902, xli., 333-350.)—The author has made a series of feeding experiments, of which the following was the most complete. The bees were fed upon 2 litres of a mixture of 2,530 grammes of dextrin syrup and 1,000 grammes of cane-sugar dissolved in 4½ litres of water. The whole of the honey was collected in two jars, the first of which contained 590 grammes and the second 1,734 grammes. On analysis the following results were obtained:

	Jar I.	Jar II.
	Per Cent.	Per Cent.
Total solids	80·8	80·17
Water	19·2	19·83
Ash	0·299	0·220
Acidity in c.c. N alkali	4·6 c.c.	4·4 c.c.
Sugar { direct	65·0	64·56
after inversion	65·96	65·28
Cane-sugar	0·91	0·67
Dextrin after fermentation	10·10	10·98
Polarization of 10 per cent. solution in 200-millimetre tube at 25° C. } direct ...	+5·7	+6·03
after inversion ...	+5·4	+5·75
Sum of dry substances	76·30	76·41

The comparative composition of the honey and of the syrup solution used was:

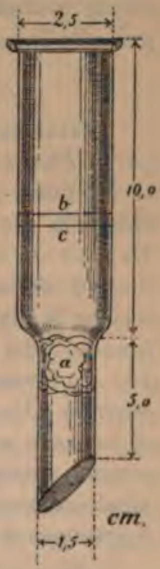
	Honey.	Syrup Solution.
	Grammes.	Grammes.
Total sugar	1519·94	804
Cane-sugar	16·97	456
Dextrin	249·98	532
Total carbohydrates	1769·92	1336

The author points out that it is remarkable that so little of the cane-sugar in the syrup remained uninverted, since in other feeding experiments on the same lines as much as 15 per cent. of cane-sugar was found in the honey. C. A. M.

Notes on the Sulphurous Acid of Beer. E. Jalowetz. (Reprint from *Mitth. Oesterr. Versuchsst. Brauerei und Malzerei*, Vienna, 1902, Heft x.; through *Chem. Zeit. Rep.*, 1902, 216.)—When sulphured and stored hops are boiled for an hour with wort in the usual manner, the amount of sulphurous acid in the latter does not increase. The sulphur of stored hops exists as sulphite. The use of sulphites in the brewery and the employment of sulphured malt increase the sulphurous acid of the beer. Freshly sulphured hops give up very little sulphurous acid to boiling water. In laboratory experiments a rise in the amount of sulphurous acid occurs during the main fermentation of wort or sugar solutions, and a decrease during the secondary fermentation; but occasionally exceptions are to be found. In three breweries investigated by the author no appreciable increase in the sulphurous acid occurred during fermentation.

It should be noted that part of the sulphurous acid of beer is combined with aldehyde. The amount is small, but as the effect of the free acid is very different from that of aldehyde-sulphurous acid, when the total acid is determined attention must be paid to the quantity which is combined. F. H. L.

Identification of Bombay Mace. P. Schindler-Zwickau. (*Zeit. für offentl. Chemie*, 1902, viii., 288-290.)—To distinguish between Bombay and Banda mace the following test is recommended: About 5 grammes of the sample are placed in the tube shown in the illustration, the lower end of the tube containing the plug of cotton-wool *a*. On gently tapping the tube, the layer of mace will settle to the line *c*. The two lines *b* and *c* serve for gauging the quantity of mace in subsequent tests. Seven to 8 c.c. of 98 to 99 per cent. alcohol are added to the tube. This quantity is absorbed by the contents of the tube. Successive portions of 7 to 8 c.c. of alcohol are then added, and each as it runs through is collected in a separate test-tube. Should the sample consist of Banda mace, on adding a drop of lead acetate to the test-tubes the first extract gives a yellow or red precipitate, the second little, and the third remains clear, or nearly so. With Bombay mace, or mixtures of both maces, the first test-tube gives a more or less red precipitate, the colour increasing in intensity in the second and third tubes, and even showing after the twenty-fifth extraction. The alcohol used must not be weaker than 98 per cent. W. P. S.



The Refractometer Number for Cod-liver Oil. Utz. (*Zeit. für öffentl. Chem.*, 1902, viii. [16], 304-306.)—The refractometer values (Zeiss' instrument) of a number of samples of cod-liver oil are given. The results for Newfoundland oils lie between 80·8 and 81·5 at 15° C., and for Norwegian oils from 82·0 to 87·5. The adulteration of cod-liver oil with paraffin oil is easily detected by the refractometer, as the value for the latter is still lower than that of Newfoundland oils.

W. P. S.

Salicylic Acid as an Adulterant in Oil of Lavender. J. E. Weber. (*Chem. Zeit.*, 1902, xxvi., 875.)—The author has met with a specimen of oil of lavender, possessing excellent odour, which gave the following constants on analysis: Specific gravity at 15° C., 0·893; rotatory power, - 6° 42′; acid value, 4·48; ester content reckoned as linalyl acetate, 35·52 per cent.; solubility, 2·5 volumes of 70 per cent.

alcohol. Apart from the acid number, which was a little high, no reason was given for assuming the oil not to be genuine; but on keeping for some time an unusual red colour developed. The colour disappeared on shaking the oil with alkali-metal hydroxide, a diminution in volume of 2·5 per cent. simultaneously taking place. It also disappeared on agitation with dilute hydrochloric acid, the aqueous extract containing iron. (This impurity was evidently derived from a bruise in the tin bottle holding the sample.) An alcoholic solution of the oil gave a dark-red colour with ferric chloride. Finally, by treating the material with potassium hydroxide, acidifying the liquid, extracting it with ether, purifying the product by repeated fractional precipitation as before, followed by crystallization from chloroform and water and decolorization with animal charcoal, white odourless crystals, melting at 156° to 157° C., were obtained, which proved to be salicylic acid. Direct tests by distillation under diminished pressure showed that no ester of salicylic acid was present. F. H. L.

Valuation of Ipecacuanha Root. G. Frerichs and N. de Fuentes Tapis. (*Arch. Pharm.*, 1902, ccxl., 401; through *Chem. Zeit. Rep.*, 1902, 254.)—Six grammes of the finely-powdered root are shaken in a dry flask with 60 c.c. of ether, 5 c.c. of ammonia are added, and after more agitation the vessel is set aside for one hour. Next 10 c.c. of water are run in, the whole is mixed up, and 50 c.c. of the ether are filtered off into a flask. Half the solvent is removed on the water-bath, and the residue is shaken out with 10 c.c. of decinormal hydrochloric acid in a separating funnel. The acid liquor is run through a paper, and the ether is washed twice with 10 c.c. of water; the combined aqueous portion is diluted to 100 c.c., and sufficient ether is added to form a layer 1 centimetre deep. Five drops of a 1 : 250 solution of iodeosin are then introduced, and the excess of free acid is titrated with decinormal potash. The volume of decinormal acid which has combined with the alkaloids, multiplied by 0·0241, gives the quantity of emetine and cephaeline in 5 grammes of the ipecacuanha root. F. H. L.

New Reaction for Quinine and Quinidine. E. Hirschsohn. (*Pharm. C. H.*, 1902, xliii., 367; through *Chem. Zeit. Rep.*, 1902, 201.)—If 10 c.c. of a neutral solution of quinine or quinidine, either as sulphate or hydrochloride, are heated to boiling with 1 drop of 2 per cent. hydrogen peroxide, a strong raspberry-red colour is produced, the intensity of which is proportionate to the amount of alkaloid present. The tint soon changes through blue-violet and blue to green. The reaction appears to be characteristic of the two alkaloids named. F. H. L.

TOXICOLOGICAL ANALYSIS.

The Guaiacum Test for Blood. **D. Vitali.** (*Giorn. di Farmac. di Trieste*, 1902, vii., 193; through *Chem. Zeit. Rep.*, 1902, 252.)—The author finds that guaiacum tincture is only coloured blue by hæmoglobin provided some turpentine oil is present, whereas all other substances which yield a similar colour oxidize the guaiacum of themselves, and do not require the turpentine. To render the test characteristic of blood, therefore, an aqueous solution of the suspected material is mixed with some fresh alcoholic tincture of guaiacum resin, and the whole is warmed to 40° or 50° C. If the mass turns blue some inorganic or animal oxidizing agent is present, and the test is not capable of showing blood; but if it remains colourless, and a blue appears on adding oil of turpentine, blood is undoubtedly to be found in the liquid examined. Ferrous sulphate behaves in the same manner as hæmoglobin, so that this salt must be absent before applying Van Deen's test. F. H. L.

ORGANIC ANALYSIS.

Estimation of Formaldehyde. **A. Pfaff.** (*Chem. Zeit.*, 1902, xxvi., 701.)—This process depends on the formation of a condensation product between formaldehyde and hydrazine, according to the equation:

$$2CH_2O + N_2H_4H_2O = C_2H_4N_2 + 3H_2O.$$

A solution of hydrazine hydrate, standardized with decinormal sulphuric acid, is added in excess to the liquid under examination, and after standing in a closed vessel for one hour, the excess of the reagent is determined by titration with the same acid, using methyl orange as indicator. The hydrazine hydrate solution does not keep its strength, but needs verification before each analysis. In standardizing it, it must be remembered that 1 molecule of sulphuric acid combines with 2 molecules of hydrazine, yielding the diammonium semisulphate $[N_2H_4]_2H_2SO_4$. The colour of the liquid obtained in this operation may be employed for comparison in the regular titration. The results quoted agree with those given by Romijn's iodine method. F. H. L.

Estimation of Anthranilic Methyl Ester. **A. Hesse and O. Zeitschel.** (*Ber.*, 1902, xxxv., 2355; through *Chem. Zeit. Rep.*, 1902, 215.)—The authors consider that the process which Erdmann has proposed (ANALYST, 1902, xxvii., 125) for this purpose has one advantage over that which they themselves have recommended (*Ber.*, 1901, xxxiv., 296), inasmuch as it does not return the methyl ester of methylanthranilic acid or any other secondary or tertiary bases which may occasionally be present in the oil; but in all other respects their own method is preferable. Erdmann's process is not quantitative, while the action of the strong acids upon the oil renders it unfit for further examination. Perhaps, when methylanthranilic acid is expected, the bases may be isolated by means of Hesse and Zeitschel's process, and the product divided, titrating one half according to Erdmann, the other in their own fashion. F. H. L.

Determination of Oil in Linseed. A. Goetzl. (*Oesterr. Chem. Zeit.*, 1902, v., 413.)—The author has studied the comparative value of petroleum spirit and ethyl ether as solvents for the oil contained in linseed. The seeds were all finely powdered, dried for two hours at 95° C. in an atmosphere free from oxygen, and their albuminous constituents were coagulated with alcohol. The extraction of the seeds is more perfect with ethyl ether, but petroleum ether yields the purer product. All the foreign substances of the oils, such as cholesterin, etc., except "linoxyn," are more soluble in ethyl ether than in petroleum ether. The colouring matter of the seeds is taken up more readily by ethyl ether. Of the two liquids petroleum ether is easier to remove from the extracted oil. F. H. L.

Some Colour Reactions of Fatty Oils. H. Kreis. (*Chem. Zeit.*, 1902, xxvi., 897.)—When Bellier's test for sesamé oil (ANALYST, 1900, xxv., 50) is carried out as he described it, but with the resorcinol replaced by phloroglucinol, no colour is produced; if, however, either hot benzene or ether is used as the solvent, the red tint develops. The best reagent is a 1 : 1,000 solution of phloroglucinol in ether shaken with equal parts of 1·40 nitric acid and the sample under investigation. So examined, arachis, sesamé, cotton, walnut, peach-kernel, castor, and perhaps other oils, give strong raspberry-red colours in the non-aqueous portion of the test; while olive oil, lard, and butter remain colourless, or yield pale orange tints. If the process is modified by moistening 0·05 gramme of solid phloroglucinol with 3 to 5 drops of oil, and then adding an equal volume of nitric acid, sesamé behaves in a very characteristic manner—the acid becomes greenish-blue, the oil red; on introducing ether it becomes violet; and on shaking with water the ether turns red-brown, and the water is stained dark-blue. This water-soluble blue dyestuff is better obtained by shaking about 0·01 gramme of phloroglucinol with 2 c.c. of a 1 : 4 solution of sesamé oil in carbon tetrachloride, adding 1 c.c. of nitric acid as before and extracting with water. Resorcinol, with or without carbon tetrachloride, gives similar reactions with sesamé, but water and ether quickly decolorize the liquids.

The author has already shown that Bellier's reaction sometimes fails altogether, or appears but faintly, after prolonged agitation, when the oils are old; and he has also found that such oils, shaken with fresh sesamé and 1·19 hydrochloric acid, give the green noticed by Bishop in the case of old sesamé (*cf.* ANALYST, 1900, xxv., 49). This peculiarity has been observed with arachis, poppy, walnut, peach-kernel, cotton, and sesamé oils; and now it appears that in the same circumstances these oils refuse to give the reaction with phloroglucinol. Thus it happens that the failure of the Bellier test does not of itself prove the purity of a specimen of olive oil, and it is necessary to try such a sample with fresh sesamé oil and hydrochloric acid to insure the non-appearance of the Bishop green.

The author points out (*Chem. Zeit.*, 1902, xxvi., 932) that when an ethereal solution of phloroglucinol is used the solution at the end of the experiment should be diluted with water; otherwise a lively disengagement of nitric oxide may take place in a short time. F. H. L.

The Analysis of Commercial Lecithins. Imbert and Merle. (*Bull. de Pharm. Sud. Est.*, May, 1902; *Ann. de Chim. anal.*, 1902, vii., 350-351.)—The amount of phosphoric anhydride in lecithins varies from 8·75 to 9·45 per cent., according to the molecular weight of the lecithin. On boiling a lecithin with a mineral acid it is decomposed into phosphoric acid, a salt of choline, and free fatty acids, whilst alkalies convert it into alkali glycero-phosphate, choline, and alkali salts of the fatty acids. The authors' method is based upon these reactions, and also on the fact that mono-alkyl phosphoric acids are mono-basic with methyl orange, but dibasic with phenol-phthalein as indicator. If, then, after decomposition of the lecithin with an acid or an alkali, the liquid be exactly neutralized, with methyl orange as indicator, the amount of phosphoric anhydride can be calculated from the amount of alkali subsequently required to effect neutralization, with phenol-phthalein as indicator; since 2 molecules of alkali correspond with 1 molecule of phosphoric acid or 2 molecules of glycero-phosphoric acid.

In applying this method about 0·5 gramme of the sample is boiled for an hour with 50 c.c. of 5 per cent. sulphuric acid and then filtered, or 0·5 gramme is treated for thirty minutes with 50 c.c. of a 2 per cent. solution of potassium hydroxide. In each case the liquid is titrated with the two indicators as described above. The method is only applicable in the absence of phosphates soluble in water or in cold acids. Further information as to the quality of a lecithin can be obtained by determining the proportion of nitrogen it contains, as the ratio of phosphorus to nitrogen is approximately $\frac{31}{14} = 2{\cdot}21$, whatever the nature of the lecithin.

C. A. M.

New Reaction for Cholesterin. E. Hirschsohn. (*Pharm. C. H.*, 1902, xliii., 357; through *Chem. Zeit. Rep.*, 1902, 201.)—On heating with distilled trichloracetic acid, cholesterin dissolves with a fine red colour. The tint gradually deepens, becoming raspberry-red in fifteen minutes, blue-violet in twelve hours, and blue after twenty-four hours. One milligramme of cholesterin treated with 10 drops of trichloracetic acid and 1 drop of acetyl chloride gives an orange colour in half an hour, which darkens to a strong cherry-red in twenty-four hours, showing a greenish-yellow fluorescence.

F. H. L.

The Composition of "Kaki-Sibu." E. Pozzi-Escot. (*Ann. de Chim. anal.*, 1902, vii., 299, 300.)—*Kaki-sibu* is a commercial antiseptic product prepared from the juice of a plant, and widely employed in Japan for coating substances with a preservative surface. The sample examined by the author was a viscous reddish-brown liquid, with a strong odour of butyric acid. It was soluble in water, the solution giving a deep blue colour with ferric chloride, and becoming rapidly brown on the addition of an alkali. On exposure to the air the sample became coated with a firm pellicle, which was insoluble in water. When distributed over a textile fabric it formed a hard, very resistant coating. On analysis it gave the following results:

Specific gravity	1·025	
Total solids dried at 105° C.	5·82	per cent.
,, ,, ,, 115° C.	5·64	,,
Ash	0·345	,,
Tannin	3·59	,,
Volatile acid (as acetic acid)	0·234	,,
Fixed acid (as lactic acid)	0·115	,,
Nitrogen	0·00	,,

The tannin was not a glucoside. C. A. M.

Determination of Oxalic Acid in Urine. W. Autenrieth and H. Barth. (*Zeits. physiol. Chem.*, 1902, xxxv., 327; through *Chem. Zeit. Rep.*, 1902, 200.)—The entire quantity of urine voided in twenty-four hours is mixed with an excess of calcium chloride solution, ammonia is added to a strong alkaline reaction, the whole is well shaken and set aside for eighteen or twenty hours. The precipitate is collected on a paper, washed with a little cold water, drained well, transferred to a beaker, and dissolved in the minimum of hot hydrochloric acid (30 c.c. of 15 per cent. acid are usually sufficient). The resulting solution is extracted by successive agitation four or five times with 150 to 200 c.c. of ether containing 3 per cent. of alcohol. The extracts are brought into a large dry flask, and allowed to rest for one hour in order to separate the last portions of water, then passed through a dry filter. Next, 5 c.c. of water are introduced to prevent the formation of the diethyl ester of oxalic acid, and the ether is distilled off, shaking the aqueous residue, if necessary, with blood charcoal, and filtering it. The product is concentrated on the water-bath to 3 or 5 c.c., treated with calcium chloride solution and excess of ammonia, allowed to stand for a time, and then faintly acidified with acetic acid. The calcium oxalate is finally collected and washed, being estimated either by ignition into oxide or by titration, the former being preferable.

The authors' experiments show them that oxalic acid is a normal, and very probably a constant, constituent of human urine. F. H. L.

The Determination of Uric Acid in Urine. E. Richter. (*Zeit. anal. Chem.*, 1902, xli., 350-359.)—The author recommends the following method, devised by Jolles, as the most exact method of determining uric acid in urine. From 10 to 15 grammes of ammonium acetate are dissolved in 100 c.c. of the clear urine, which is then rendered faintly alkaline with ammonia, and allowed to stand for five to six hours. The precipitated uric acid is then collected on a filter, and washed with a 10 per cent. solution of ammonium carbonate until the filtrate is free from chlorine. The precipitate is then washed by means of hot water into a beaker, and boiled for about an hour with 0·1 gramme of magnesia to remove the excess of ammonium carbonate, after which 10 c.c. of sulphuric acid (specific gravity 1·4) are introduced, and the liquid oxidized by means of a 0·8 per cent. solution of potassium permanganate. This is first added to the boiling liquid 1 c.c. at a time until it is no longer rapidly decolorized, and then only 6 to 8 drops are added. When the separated manganese peroxide no longer dissolves in the liquid when evaporated from about 500 c.c. to 100 c.c. (not less), there can be no doubt as to the whole of the uric acid having been oxidized to urea. The liquid is now evaporated to about 30 c.c., and any manganese peroxide dissolved by the addition of a few drops of $\frac{N}{10}$ oxalic acid, after which the beaker is immersed in cold water and continually agitated, whilst sodium hydroxide solution (32° Bé) is added 1 c.c. at a time until the reaction becomes alkaline to litmus. The liquid is then transferred to a Reichert's nitrometer, and shaken with 25 c.c. of a solution prepared by dissolving 80 grammes of sodium hydroxide in water, adding 25 grammes of bromine, and diluting the liquid to a litre. The amount of nitrogen liberated is corrected for temperature and pressure, and calculated into the corresponding amount of uric acid.

In this way the author obtained results ranging from 98·5 to 100·3 per cent. of the amount of uric acid used in test experiments. As a rule, the results given by this method were from 1 to 2·5 per cent. higher than those yielded by the Ludwig-Salkowski method, which, however, has been admitted by its authors to give results about 2 per cent. too low.

C. A. M.

The Oxidation and Estimation of Uric Acid and Urates. J. F. Tocher. (*Pharm. Journ.*, 1902, 161-166.)—As urates are quantitatively converted into urea by boiling with dilute sulphuric acid and chromic anhydride, the uric and urea nitrogen can be determined in urine as follows: A measured volume of the urine is saturated with solid ammonium chloride, and the ammonium urate collected on a filter and washed with ammonium chloride solution. The urate is then dissolved in dilute soda solution, and boiled to drive off the ammonia; after acidifying with dilute sulphuric acid 2 to 3 grammes of chromic anhydride are added, and the solution is boiled. It is then cooled and transferred to a nitrometer, and the nitrogen liberated with hypobromite as usual. One c.c. of nitrogen is equivalent to 0·00375 gramme of uric acid. An alternative method is to boil 10 c.c. of the sample with about 2 c.c. of dilute sulphuric acid and 2 grammes of chromic anhydride for two minutes. The solution is then cooled and made up to 10 c.c.; 1 c.c. of this is measured into a hypobromite tube, and the liberated nitrogen read off. On separately estimating the urea in 1 c.c. of the urine, from which the urates have been precipitated as barium urate, and deducting the amount found from that obtained by the chromic anhydride treatment, the difference multiplied by 1·4 gives the uric acid.

W. P. S.

A New Method of Organic Analysis. P. Thibault and A. C. Vournasos. (*Bull. Soc. Chim.*, 1902, xxvii., 895-901.)—It is stated that by means of the apparatus devised by the authors all the disadvantages of the ordinary method of combustion are obviated, and several analyses can be made in a day. It consists of a vertical steel tube, 20 centimetres in length and 5 centimetres in diameter, terminating below in a spherical bulb. On the side of the tube near the top is brazed a narrow steel tube, bent once at a right angle, and provided outside with a tap for the introduction of oxygen. The other end of this tube passes down into the bulb of the combustion tube. The top of the latter is provided with a collar, into which a cap can be fitted and hermetically closed, whilst a narrow steel tube leads from the cap and is connected with the ordinary absorption bulbs. The tube is heated from the upper part downwards by means of a blowpipe. When the upper part of the tube becomes bright red the lower part is heated by means of a second blowpipe.

A weighed quantity of the substance is introduced, and the tube filled with cupric oxide, which is pressed down as much as possible. The cover is then fixed on and connected with the absorption bulbs, and the tube heated as described above. As soon as bubbles of gas cease to be evolved the tap of the side tube is opened, and a slow current of oxygen passed through for about fifteen minutes. In the case of readily combustible bodies the combustion is complete within an hour, whilst for less combustible bodies the time is slightly increased, but in every instance the results are stated to be much more exact than those obtained by other methods. The apparatus can also be used for the combustion of liquids and gases. In the case of volatile liquids a weighed quantity of the substance is placed in a tube provided with a tap at each end, one being connected with the side tube of the apparatus, whilst a current of oxygen, heated by passing through a red-hot tube, enters by the other. The tube is first heated, and when the cupric oxide becomes red-hot the mixture of liquid and hot oxygen is slowly introduced. A similar plan is adopted for the combustion of gases, but in this case the oxygen is used at the ordinary temperature. C. A. M.

INORGANIC ANALYSIS.

Mercury Assay. R. Biewend. (*Berg. u. Hüttenmänn. Zeit.*, 1902, lxi., 441; through *Chem. Zeit. Rep.*, 1902, 251.)—The author has modified Eschka's dry method of determining mercury as follows: The sample is finely ground, dried over sulphuric acid, and a quantity is taken which does not contain more than 0·15 gramme of mercury. This is covered with a layer of copper filings, 0·3 to 0·5 centimetre deep in a porcelain crucible, and the whole is covered with magnesia, or kieselguhr and asbestos. The gold basins weigh 10 to 12 grammes each, and have a cavity 3·5 to 4 centimetres in diameter by 0·75 centimetre deep, with a rim 0·5 centimetre wide. The crucible is supported by means of a perforated piece of asbestos sheet, so that the edges of the gold basins are protected from the heat. The crucible is first heated gently for ten minutes with a spirit-lamp; then the gold basin is changed for another, and ignition is continued for ten minutes at a higher temperature, which makes the bottom of the crucible appear red. The cooling water in the basins is renewed during the operation. F. H. L.

Volumetric Estimation of Gold with Thiosulphate. F. Faktor. (*Casopis pro prumysl chemicky*, 1902, vi., 183; through *Chem. Zeit. Rep.*, 1902, 200.)—If a neutral solution of gold chloride is treated with an excess of potassium iodide, the original green precipitate of gold iodide redissolves, yielding a brown liquid which contains AuI_4K. On adding to this slowly a dilute solution of thiosulphate, the double iodide loses two atoms of iodine, becoming AuI_2K. On this reaction the author bases his process, using 4 grammes of potassium iodide, decinormal thiosulphate, and starch as indicator.

F. H. L.

A Colorimetric Method of Determining Arsenious Acid. J. Mai. (*Zeit. anal. Chem.*, 1902, xli., 362-365.)—The author's method is based on the conversion of the arsenic compounds into arsenious chloride, and of the latter into arsenic trisulphide. The determination is carried out in an apparatus similar to that employed by Wiborgh for the determination of hydrogen sulphide. In preparing standard deposits, the arsenic trioxide was cautiously heated with hydrochloric acid (specific gravity 1·19) in a large test-tube and the arsenious chloride slowly distilled over, whilst a current of dry carbon dioxide was simultaneously passed through the apparatus. The gases passed through a U-tube and then into a wide-mouthed tube, the open end of which was covered with moist cotton impregnated with zinc sulphide. The cotton cloth had been soaked in zinc sulphate solution and then subjected to the action of hydrogen sulphide. It was fixed to the mouth of the tube by means of a rubber ring, and the tube itself was immersed in water saturated with fresh hydrogen sulphide. In order to prevent the cloth being blocked, a few pinholes were made, which, however, did not allow any of the arsenic to pass through. The mirrors given by quantities of arsenic, ranging from 0·1 to 0·6 milligramme, showed marked differences between each other, but above 0·6 milligramme the increase in intensity was only perceptible after the addition of 0·2 milligramme.

C. A. M.

The Quantitative Determination of Small Quantities of Arsenic. C. T. Mörner. (*Zeit. anal. Chem.*, 1902, xli., 397-413.)—The method is based upon the oxidation of arsenic trisulphide by means of alkaline permanganate,

$$5As_2S_3 + 28KMnO_4 + 27SO_3 = 5As_2O_5 + 14K_2SO_4 + 28MnSO_4,$$

according to which 1 c.c. of $\frac{N}{100}$ potassium permanganate solution is equivalent to 0·0536 milligramme of arsenic. In the titration the arsenic trisulphide is dissolved in alkali (0·5 per cent. potassium hydroxide solution) and treated with 25 c.c. $\frac{N}{100}$ permanganate solution. After shaking the flask, 5 c.c. of 5 per cent. sulphuric acid are introduced, the liquid heated until colourless, and titrated back with $\frac{N}{100}$ permanganate solution.

In applying this method to the determination of arsenic in fabrics, such as carpets, etc., the sample is distilled with concentrated hydrochloric acid, and the distillate received in dilute nitric acid and evaporated to dryness, or the distillate is collected in water, the arsenic precipitated with hydrogen sulphide, the precipitate dissolved in ammonium hydroxide, and the solution evaporated to dryness. In either case the residue will contain organic substances capable of oxidation. To eliminate these the dry residue is heated on the water-bath, and treated successively with 2 c.c. of 0·5 per cent. potassium hydroxide solution; then, after about one minute, with 2 c.c. of 5 per cent. permanganate solution and heated for three minutes; then with 2 c.c. of 5 per cent. sulphuric acid and heated for three minutes;

and, lastly, with 1 c.c. of 20 per cent. tartaric acid solution and heated until colourless. The liquid is now filtered into another small basin, the first basin being rinsed with 2 c.c. of water. The second basin is placed on the water-bath, and after about one minute 1 c.c. of 5 per cent. thio-acetic acid is introduced, and the basin heated for three minutes, and left to cool for about five minutes. The precipitated arsenic trisulphide is collected and washed twice with 5 c.c. of 0·5 per cent. sulphuric acid, and then three times with 2 c.c. of water. It is then washed by means of three successive portions of 2 c.c. each of 0·5 per cent. potassium hydroxide solution into a flask containing $\frac{N}{100}$ permanganate solution, and the arsenic determined as described above, with the addition that a correction of 0·3 c.c. is made for substances extracted from the filter-paper by the alkali.

The author gives a detailed account of the results of forty test experiments in which small quantities of arsenic (0·17 to 0·25 milligramme) were added to different fabrics, etc. The differences between the amounts taken and found varied from −0·03 to +0·04 milligramme.

C. A. M.

Use of Sulphuric Acid as a Solvent for Minerals rich in Arsenic, Iron, and Lead. H. Nissenson and F. Crotogino. (*Chem. Zeit.*, 1902, xxvi., 847.)—The authors call attention to the great superiority of suphuric acid over nitric or hydrochloric acid for the dissolution of substances containing a large proportion of arsenic or lead. In the first place, conversion of the arsenic and antimony into their more highly oxidized state is avoided, so that the subsequent precipitation with sulphuretted hydrogen is easier to manage; and, in the second place, danger of losing arsenic by volatilization of arsenious chloride is prevented. The material, finely ground and dried at 100° C., is heated with three times its weight of strong sulphuric acid for thirty minutes to three hours—*i.e.*, till everything which is soluble has been attacked; the liquid is then cooled quickly, diluted with hot water (not cold), allowed to settle, and filtered. In presence of antimony the residue is extracted with ammonium tartrate, and the gangue is filtered off; the lead is precipitated with a further amount of sulphuric acid, while any antimony in the filtrate therefrom is added to the bulk solution obtained after treatment of the diluted sulphuric acid solution with sulphuretted hydrogen. In their original article the authors give full details as to the adaptation of their methods to various ores, such as copper and iron pyrites, arsenical pyrites, lead ores, particularly lead glance, and minerals containing zinc and lead. Zinc ores containing much lead or iron may be very conveniently analysed or arsenic and antimony by the same process.

F. H. L.

Determination of Tin, and its Separation from Antimony. C. Ratner. (*Chem. Zeit.*, 1902, xxvi., 873.)—The author recommends with Rössing (ANALYST, 1902, xxvii., 99), Clark's method as being the only accurate one for separating tin from antimony; but the second precipitation of the antimony sulphide advocated by Rössing is not necessary, provided the original solution was not too concentrated—*i.e.*, did not contain more than 0·5 gramme of tin plus antimony per 600 or 700 c.c.

Both Clark's and Rössing's processes for the estimation of the tin in the antimony filtrate are objectionable, and the following method is preferable: The sulphuretted hydrogen is driven out of the liquid, and a fragment of pure zinc, as compact as possible, is dropped into the beaker, which is heated over gauze without permitting its contents to boil for some twenty minutes till a qualitative test shows that all tin has been thrown out of solution. The metallic residue is then collected on a paper, whence it is rinsed back into a small beaker with about 15 c.c. of water. Here it is treated with 10 c.c. of nitric acid, the temperature being adjusted to cause smooth dissolution. When all the zinc has disappeared the volume of the liquid is made up to 40 or 50 c.c., the precipitate is boiled up, allowed to settle, washed by decantation, and finally thrown on a paper, dried, ignited, and weighed. As metastannic acid filters badly, some ammonium nitrate may be added to the solution. The zinc occasionally becomes covered with a glass-like crust, but this does not appear to happen until all the tin has been deposited; and if it be formed the analysis is not affected. Three examples are given in which the figures obtained by the present method agree well with those of Rössing's and Clark's processes.

F. H. L.

Detection of Cadmium and Zinc by Blowpipe Tests. R. Biewend. (*Berg. u. Hüttenmänn. Zeit.*, 1902, lxi., 413 and 425; through *Chem. Zeit. Rep.*, 1902, 251.)—When an ore containing less than 1 per cent. of cadmium is heated before the blowpipe the usual brown sublimate of cadmium oxide fails to appear. In certain circumstances, however, a dull black, soot-like ring is produced, blue or violet at its inner edge, and this ring develops before the zinc sublimate. Antimony, bismuth, a few of the rare metals, and especially lead, interfere; otherwise the test shows 0·002 gramme in 1 gramme of substance. It indicates cadmium in brass foil. When zinc ores or alloys are heated in a glass tube before the blowpipe, using dry potassium oxalate as a reducing agent if necessary, cadmium yields a silver-white mirror of metal and a brown sublimate of oxide, which appears gray while hot. Lead, bismuth, and tin give no sublimates, and antimony only if it exists as sulphide. Full details are given respecting this test in the original article. F. H. L.

Separation and Precipitation of Thorium. A. Kolb. (*J. prakt. Chem.*, 1902, lxvi., 59; through *Chem. Zeit. Rep.*, 1902, 214.)—In order to precipitate the hydroxide from a solution of pure thorium nitrate, the liquid is acidified with a little hydrochloric or nitric acid, neutralized as far as possible with ammonia, and raised to the boiling-point. The boiling liquid is then treated, drop by drop, with aniline, shaking vigorously until a turbidity appears. The vessel is next put aside in a warm place till the precipitate settles, and the latter is washed by decantation with water at 50° C. Commercial thorium sulphate should first be mixed with ammonia, the hydroxide dissolved in hydrochloric acid, the solution neutralized with ammonia, and further treated as above, any ferric oxide or ceric oxide being reduced with sulphurous acid or sulphuretted hydrogen before adding the aniline. A solution of the chlorides or nitrates is most suitable for the separation of thorium by this method. F. H. L.

Iodometric Valuation of the Peroxides of Barium, Sodium, etc. **E. Rupp.** (*Arch. Pharm.*, 1902, ccxl., 437; through *Chem. Zeit. Rep.*, 1902, 251.)—The peroxides of the alkalies and alkaline earth metals may be valued by treating them with potassium iodide in presence of a little hydrochloric acid, and titrating the liberated iodine with decinormal thiosulphate and starch. The following is the reaction which takes place: $M''O_2 + 4HCl + 2KI = M''Cl_2 + 2KCl + 2H_2O + I_2$. F. H. L.

The Detection of Bromine and Iodine in the Presence of Thiosulphates. **A. F. Leuba.** (*Ann. de Chim. anal.*, 1902, vii., 298, 299.)—The solution of the salt is heated to the boiling-point, and the thiosulphate precipitated by means of a slight excess of lead nitrate. The liquid is boiled for fifteen minutes and then filtered, and the filtrate tested for iodine by the addition of chlorine water and carbon bisulphide. The lead iodide formed in the precipitation of the thiosulphate is sufficiently soluble in boiling water to give the violet reaction in the filtrate. For the detection of bromine, it is preferable to add a little fluorescein to the liquid rendered alkaline with sodium hydroxide, when a bright red colour is obtained in the presence of bromides.

C. A. M.

The Volumetric Determination of Iodides in the Presence of Bromides and Chlorides. **Thomas.** (*Comptes Rendus*, May 20, 1902; *Ann. de Chim. anal.*, 1902, vii., 349, 350.)—When a solution of a thallic salt, such as the chloride, is mixed with an excess of potassium iodide, a greenish-black precipitate, consisting of a mixture of thallous iodide and iodine, is formed. If, however, the thallic salt is in excess, only free iodine is liberated; and on this fact is based the author's method, in which the iodine is expelled from the liquid by boiling, and the unreduced thallic salt determined by titration. For this purpose the solution is mixed with an excess of standard thiosulphate solution and of potassium iodide (free from iodate) and 1 c.c. of starch-water, and titrated with a standard solution of iodine.

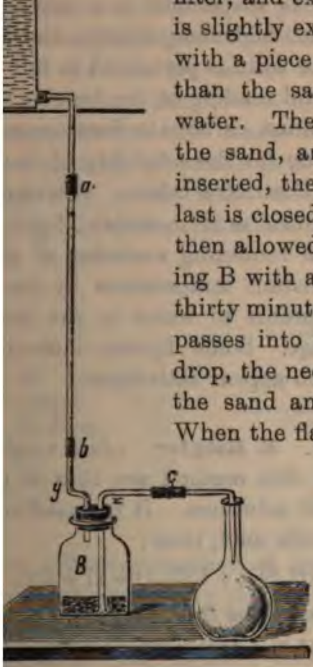
In applying the method in the presence of bromides, the liquid must not be overheated, but the free iodine expelled by means of a current of air.

The precipitated thallous iodide can readily be converted into thallic chloride by treatment with hydrochloric acid and potassium chlorate. After removal of the chlorine by heating, the liquid is diluted, and gives a solution of thallic chloride which can be at once used again. There is practically no loss of thallium.

C. A. M.

The Titration of Free Alkali in the Presence of Nitrites. **K. Arndt.** (*Zeit. anal. Chem.*, 1902, xli., 359-362.)—If the solution of the nitrite be sufficiently cooled and a suitable indicator employed, the alkali can be titrated with standard acid. Thus, on cooling a solution of 1 gramme of sodium nitrite to 0° C., and titrating the liquid with $\frac{N}{10}$ sulphuric acid with methyl orange as indicator, there was hardly any perceptible odour of nitrogen peroxide, nor did the liberated nitrous acid turn the indicator red. Litmus as indicator gave still better results, although the change in as not so sharp as in the absence of nitrites. If carbonates be present, it is y to precipitate them with barium chloride, and to titrate the filtrate with l hydrochloric acid. In the case of aurin (rosolic acid) the change is sharper the case of litmus, but, since nitrous acid acts upon alcohol, it is necessary oy an aqueous solution of the indicator. Here, too, carbonic acid must be l before the titration. This method gave concordant results in determining unt of free ammonia in a solution of ammonium nitrite. C. A. M.

termination of Prussian Blue in Spent Oxide. R. Schwartz. (*Chem. Zeit.* vi., 874.)—The apparatus employed by the author for extracting spent gas-xide with sodium hydroxide and carbonate is shown in the annexed sketch. B is a wide-necked bottle holding 100 to 150 c.c. and containing a layer 1·5 to 2 centimetres thick of coarse washed sand free from dust. The tube *x* passes right through the sand filter, and extends almost to the bottom of the bottle, where it is slightly expanded trumpet-fashion, having its orifice covered with a piece of brass or phosphor-bronze gauze finer in mesh than the sand particles. A is a pressure vessel filled with water. The weighed quantity of the sample is spread out on the sand, and the extracting fluid is added; the stopper is inserted, the rubber connections *a*, *b*, and *c* are made, and the last is closed with a screw clamp. Maceration in the cold is then allowed to proceed for one or two hours, or, by surrounding B with a water-bath, digestion in the hot is continued for thirty minutes. The clamp *c* is next opened so that the liquid passes into the 1-litre measuring flask on the right, drop by drop, the necessary pressure to determine percolation through the sand and gauze being given by the head of liquid in *y*. When the flask is full to the mark a few drops of liquid are collected in a test-tube, and examined for alkalinity and for ferrocyanogen. Generally the liquid is neutral and free from dissolved matter; if not, more water, or more sodium hydroxide, may be introduced through A and a second litre of extract collected. Finally, the ferrocyanides in the solution are determined as usual, preferably by gravimetric methods. The process sults agreeing well with those of Knublauch's, and Moldenhauer and Leybold's s; but it has the advantage of yielding a clear extract, and of showing when ble matter has been recovered. F. H. L.



e Behaviour of Selenious Acid in the Marsh Apparatus. J. Schindel- (*Zeit. für öffentl. Chem.*, 1902, viii. [16], 306-309.)—The addition of

0·25 gramme of selenious acid to a Marsh apparatus containing 10 grammes of pure zinc and about 100 c.c. of sulphuric acid (1 : 5) quickly caused the evolution of hydrogen to cease. A brown-red flocculent precipitate formed in the solution, and was found to consist of a mixture of selenium and zinc containing from 11 to 19 per cent. Zn. No selenium passed over into the combustion tube. When aluminium and soda were employed, selenious acid did not hinder the evolution of hydrogen. In the presence of arsenic (10 to 25 c.c. of a 0·05 per cent. solution of As_2O_3) no mirror was obtained until the selenious acid had been converted into the brown flaky condition. The arsenic then came over, none remaining with the zinc-selenium compound.
W. P. S.

Use of Victor Meyer's Vapour Density Apparatus for Gas-volumetric Analysis. J. Mai and M. Silberberg. (*Chem. Zeit.*, 1902, xxvi., 875.)—This is a preliminary note suggesting an employment of V. Meyer's apparatus for the analysis of such substances as give off a gas on treatment with some liquid reagent. The latter is placed in the inner tube, and the solid is suspended in a bag of ignited asbestos above it. The apparatus is raised to some high temperature, and the level of the liquid in the burette is read off; then the solid is permitted to fall into the liquid, and when all gas has been evolved a second reading of the burette is taken. Experiments were first carried out with pure sodium carbonate decomposed in the cold with hydrochloric acid, correction being made for the solubility of the carbon dioxide in the water of the burette by means of Dietrich's tables. Afterwards the carbonate was decomposed with hot strong sulphuric acid to which 15 per cent. of sodium sulphate had been added with the view of rendering evolution of gas more gentle, the apparatus being maintained at a constant temperature by the use of boiling amyl acetate in the jacket. Oil is preferable to water in the measuring vessel, and mercury may be used with advantage. Some figures indicating the determination of carbon dioxide are recorded, which appear satisfactory. F. H. L.

A Gas-Volumetric Method of Alkalimetry. E. Riegler. (*Zeit. anal. Chem.*, 1902, xli., 413-419.)—The advantages claimed for this method are that it is rapid, very accurate, and obviates the use of all standard solutions. It is based upon the fact that hydrazine sulphate reduces the salts of iodic acid, thus:

$$(1)\ 5N_2H_4.H_2SO_4 + 4NaIO_3 = 2Na_2SO_4 + 3H_2SO_4 + 12H_2O + 4I + 5N_2.$$

The excess of hydrazine sulphate present then acts on the free iodine:

$$(2)\ N_2H_4.H_2SO_4 + 4I + 2H_2O = H_2SO_4 + 2H_2O + 4HI + N_2.$$

Hence the decomposition of an iodate by an excess of hydrazine sulphate can be represented by the equation,

$$(3)\ 6N_2H_4.H_2SO_4 + 4NaIO_3 + = 2Na_2SO_4 + 4H_2SO_4 + 4HI + 12H_2O + 6N_2;$$

and, according to this, 1 milligramme of nitrogen corresponds with 4·175 milligrammes of iodic acid, or 1 c.c. of nitrogen of 0° C. and 760 millimetres' pressure is equivalent to 5·2216 milligrammes of iodic acid. If, then, the equivalent quantity of a given base combining with iodic acid be known, its quantity can be calculated from the amount of nitrogen. This weight, x, can be found by multiplying the volume of nitrogen at 0° C. and 760 millimetres pressure by the factor 0·0297, and multiplying the product by the equivalent of the base in question.

In making a determination the base is dissolved in 20 to 30 c.c. of water. After the addition of 2 to 3 drops of phenolphthaleïn solution a 2 per cent. solution of iodic acid is introduced drop by drop with continual shaking until the liquid becomes colourless. The solution is then placed in the exterior vessel of a Knop-Wagner nitrometer, whilst about 0·5 gramme of hydrazine sulphate is placed in the inner vessel, and the nitrogen liberated and measured in the usual manner. It is essential to shake the nitrometer flask during the evolution of the gas at intervals for about thirty seconds at a time until its contents become colourless. C. A. M.

The Determination of the Oxygen Absorption of Natural Waters. L. W. Winkler. (*Zeit. anal. Chem.*, 1902, xli., 419-426.)—The loss of oxygen on boiling 100 c.c. of acid $\frac{1}{100}$ N-permanganate solution was found by the author to be equivalent to 0·125 c.c., whilst in the case of an alkaline solution the average loss was 0·175 c.c. In the author's opinion, an alkaline solution is preferable, as the organic substances are more completely oxidized, and chlorides have a less disturbing influence. He has also made experiments to determine under what conditions a standard solution of oxalic acid will keep best, and finds that that acidified with sulphuric acid is the most satisfactory. Thus, the following comparative results were obtained, the initial values being taken as 100 :

	$\frac{1}{100}$ N-Oxalic Acid Solution.	$\frac{1}{100}$ N-Oxalic Acid Solution Neutralized.	$\frac{1}{100}$ N-Oxalic Acid Solution Acidified.	$\frac{1}{100}$ N-Oxalic Acid Solution rendered Alkaline.
Initial values	100·00	100·00	100·00	100·00
After 35 days	99·47	96·21	99·94	99·49
„ 61 „	99·24	93·92	99·89	98·87
„ 90 „	98·75	92·52	99·74	98·18
„ 183 „	98·34	87·87	99·69	96·07

The following method of determining the oxygen absorption is recommended: 100 c.c. of the water are kept boiling for ten minutes with 10 c.c. of $\frac{1}{100}$ N-alkaline permanganate, prepared with the usual precautions and standardized on $\frac{1}{100}$ N-oxalic acid solution. The liquid is then treated with 10 c.c. of dilute sulphuric acid (1 : 3) and 10 c.c. of $\frac{1}{100}$ N-oxalic acid solution, and, after it becomes completely colourless, the alkaline permanganate solution is introduced, drop by drop, from a burette until a faint pink colour remains. A correction of 0·3 c.c. is made, and the oxygen absorption expressed in terms of c.c. of $\frac{1}{100}$ N-permanganate solution reduced by 100 c.c. of the water. C. A. M.

APPARATUS.

Platinum Basin with Chimney and Draught-holes. A. Hebebrand. (*Zeit. für Untersuch. der Nahr. und Genussmittel,* 1902, v., 719-721.)—The apparatus consists of a platinum basin having a number of circular holes around and just below its edge. The basin is covered with an aluminium lid, from the centre of which a chimney rises. The latter is about 11 centimetres high and 1·8 centimetres in diameter. The top of the chimney is closed, the opening being at the side, so that no dust, etc., may fall into the basin. Substances which burn with difficulty, such as meat, bread, and sugar, are completely incinerated in about one-half the time usually required, and the amount of ash agrees closely with that obtained by igniting in an open basin. W. P. S.

Apparatus for Cold and Hot Filtrations. P. N. Raikow. (*Chem. Zeit.,* 1902, xxvi., 732.)—In the annexed sketches are shown jackets for surrounding funnels with either freezing mixtures or steam, which have the advantage over other devices that not only the sides, but also the top of the liquid being filtered is directly exposed to the desired temperature conditions. In the cold funnel (Fig. 1) the vessel *b* is a hollow dish with depending flange fitting round the jacket, and is filled with ice or freezing mixture. The inner funnel should be fixed as high as possible in the jacket, and the annulus filled completely, so that air-space may be reduced to a minimum. In the hot apparatus (Fig. 2) *c* is a coned cover connected at *p* with the steam-supply, and joined at *u* by means of the rubber tube *k* with the inlet of the lower jacket. If preferred, *k* may be done away with by making *u* and *r* vertical so as to form a telescopic joint. F. H. L.

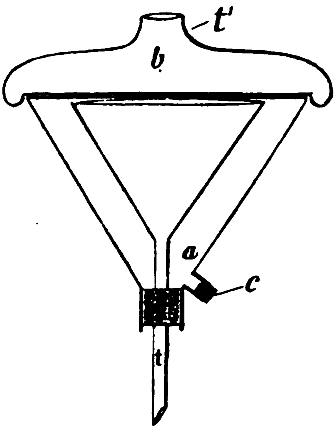


FIG. 1.

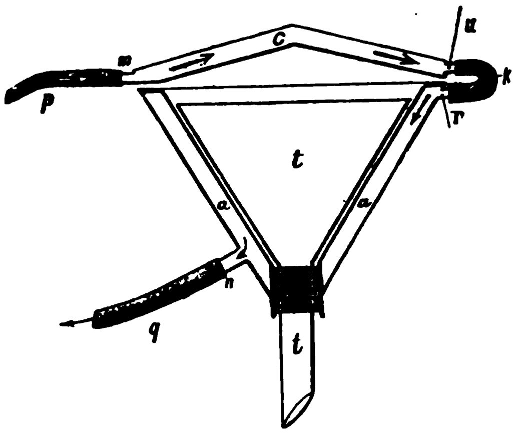
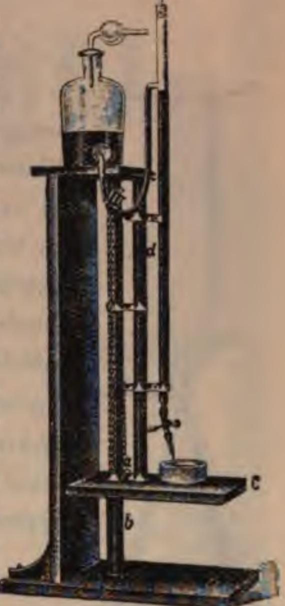


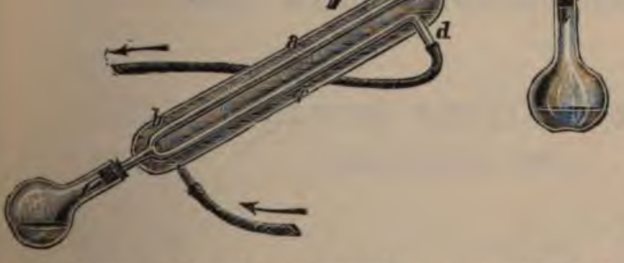
FIG. 2.

Automatic Apparatus for Titrations. W. Schmidt. (*Chem. Zeit.*, 1902, xxvi., 734.)—This is a burette stand which automatically sets the burette at its zero-point, and is much simpler than most other patterns, avoiding the usual inconvenient rubber ball. The burette itself is clamped on to the rod *d*, which is rigidly connected with the little table *c*; *c* and *d* together slide up and down the rod *b* over a distance of about 14 centimetres. The chain shown in the diagram is fastened at one end to *c*, passes over a pulley at the top of *b*, and, when the table is at its highest position, is hooked over a pin *a* on the table on the further side. When not in use *c* is allowed to rest on the base of the main stand, in which position the zero-point of the burette is 2 centimetres below the lowest position of the liquid in the store bottle, and the reagent flows into the burette to fill it. When the small table is raised the zero-point is about 2 centimetres above the highest position of the liquid in the store vessel, and the reagent runs back through the curved rubber tube till the O-point is set correctly. The burette cannot overflow, as it is some 12 centimetres longer than the zero-spot. The elevated position of the small table is specially convenient during titration, since it renders the pinchcock or stopcock more accessible than usual. The apparatus is made by J. Brüncker and Co., of Ilmenau.



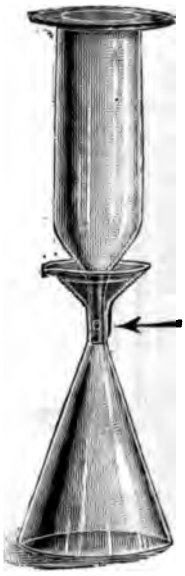
F. H. L.

Condenser for Volatile or Noxious Liquids. H. Goeckel. (*Chem. Zeit.*, 1902, xxvi., No. 55.)—This is an apparatus of the Liebig pattern, in which the inner tube is forked at the outlet end, one branch passing through a cork to the receiving flask, and the other returning backwards to the opposite extremity, where it is bent and passed through the outer wall. By this arrangement the condensing surface for a given length of water-jacket is doubled; while the tube *d* may either be connected to a second condenser when extremely volatile liquids are being distilled, or it may be coupled to a pump for distillations under diminished pressure. If the vapours are unpleasant or poisonous, the rubber tube fastened to *d* can be led into the draught cupboard. The tube *a* is shown as lying vertically over *e* for convenience of illustration, but actually they are twisted so as to



be in a horizontal plane. The apparatus is protected as a "Gebrauchsmuster," and may be obtained from Sauer and Goeckel, 49, Wilhelmstrasse, Berlin, W.

F. H. L.



A Modified Wiborgh Flask. H. Goeckel and J. Wolfmann. (*Stahl und Eisen*, 1902, No. 12.)—As shown by the illustration, the Erlenmeyer flask in this apparatus is expanded at its neck so as to form a funnel, while at the spot indicated by the arrow it is provided with a vertical channel. The lower extremity of the upper vessel is also provided either with a similar and opposite channel, or with a hole in its neck, through which liquid can be introduced into the flask. This device has the advantage of doing away with any glass stop-cock, and of making the apparatus less liable to injury. The joint may be made perfectly tight by pouring water into the funnel. The apparatus may be obtained from Sauer and Goeckel, Berlin.

F. H. L.

INSTITUTE OF CHEMISTRY.

Examinations of the Institute will be held in London during January, 1903. The Intermediate Examination will commence on January 6. Examinations on all branches of the Final, excepting Biological Chemistry, will commence either on January 6 or January 13. Applications for admission to the examinations must be forwarded to the Registrar not later than Tuesday, December 2, 1902.

NOTICE TO SUBSCRIBERS.

Council of the Society have made fresh arrangements with regard to the future publication f The Analyst, and as these will not come into operation until January 1, 1903, they will be d to subscribers if they will, until the end of this year, forward subscriptions to the Hon. urer of the Society.

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VOLUME XXVII.—No. 321.
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THE ANALYST.

DECEMBER, 1902.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE monthly meeting of the Society was held on Wednesday evening, November 5, in the Chemical Society's Rooms, Burlington House. The President, Dr. J. Augustus Voelcker, M.A., B.Sc., occupied the chair.

The minutes of the previous meeting were read and confirmed.

A certificate of proposal for election to membership in favour of Mr. P. Macdonald was read for the second time; and certificates in favour of Messrs. Alexander Hutcheson Bennett, analytical chemist, 39, Lime Street, London, E.C.; James Charles McWalter, M.A. (R.U.I.), D.P.H. (Dublin), analytical chemist, 19, North Earl Street, Dublin; and William Partridge, A.I.C., 2, Ethelbert Road, Wimbledon, assistant to Mr. William Chattaway, F.I.C., were read for the first time.

The following papers were read: "The Reactions of the Alkaloids of Ipecacuanha," by Alfred H. Allen and G. E. Scott-Smith; "The Analysis of Preparations containing Opium," by Alfred H. Allen and G. E. Scott-Smith; "Note on the Estimation of Salicylic Acid," by Sidney Harvey; and a "Note on the Volatility of Aqueous Solutions of Acetic Acid," by William Chattaway.

CERTAIN REACTIONS OF THE ALKALOIDS OF IPECACUANHA.

BY ALFRED H. ALLEN AND G. E. SCOTT-SMITH.

(*Read at the Meeting*, *November* 5, 1902.)

OUR attention was recently called to the fact that the alkaloids of ipecacuanha in some of their colour-reactions presented a considerable similarity to morphine, and we have made a number of experiments with the view of ascertaining to which of the ipecacuanha alkaloids these colour-reactions were attributable.

The best recent work on the alkaloids of ipecacuanha is that of Dr. B. H. Paul and A. J. Cownley. They have recognised, and isolated from ipecacuanha root, three alkaloids — namely, emetine ($C_{30}H_{44}N_2O_4$), cephaëline ($C_{28}H_{40}N_2O_4$), and psychotrine—to the last of which no formula has hitherto been assigned. It does not, however, follow that these are the only alkaloids present in ipecacuanha root, and in the opinion of a high authority there are at least five such bodies present. However, our experiments have been practically limited to the three alkaloids recognised by Paul and Cownley.

Of these alkaloids of ipecacuanha emetine is present in the largest amount, but a considerable proportion of cephaëline is also present. Psychotrine occurs in much smaller quantity. All three alkaloids are soluble in amylic alcohol. Emetine, which appears to be an amorphous alkaloid, yields crystallizable salts. Cephaëline crystallizes well, and psychotrine also forms highly characteristic crystals when properly treated. Emetine and cephaëline, when freshly precipitated, differ from psychotrine in being readily dissolved by ether after adding excess of ammonia to their salts, whereas psychotrine is not so extracted, but may be dissolved by subsequently agitating the ammoniacal liquid with chloroform.* Dry ether has little solvent action on dry emetine or cephaëline. Emetine and cephaëline are separated by Paul and Cownley by adding excess of caustic soda to the solution of their salts and agitating with ether, which takes up the emetine only. In this behaviour cephaëline closely resembles morphine.† The cephaëline can be thrown down from the alkaline liquid by adding chloride of ammonium, when it separates in crystals, or the soda may be neutralized with acid, ammonia added, and the cephaëline removed by agitation with ether. These processes must be repeated several times to insure a perfect separation of the alkaloids. We extracted the alkaloids from a sample of Carthagena ipecacuanha root, kindly sent us by Mr. W. Chattaway, by moistening the ground root with ammonia and percolating with amylic alcohol, this process being recommended by Mr. A. J. Cownley. The alkaloids extracted had the following percentage composition :

Emetine	... ...	50·9 per cent.
Cephaëline	... ...	43·8 ,,
Psychotrine	... ...	5·3 ,,

The alkaloids isolated by the foregoing method were assumed to be pure, but we believe this was not strictly the case.

The alkaloids were isolated by the same process from a sample of Brazilian (Rio) ipecacuanha root, but the relative proportions were not ascertained.

We have also isolated the mixed alkaloids from several samples of liquid extract of ipecacuanha (British Pharmacopœia), and separated the alkaloids from one of these, without determining the amount, for the examination of their colour-reactions.

The process employed for the extraction of the mixed alkaloids was to evaporate the alcohol, dilute the residue with water, acidulate the liquid with hydrochloric acid, and agitate with amylic alcohol. This treatment would remove any glucosides present and other bodies of a non-alkaloidal nature. The aqueous liquid was then treated with sodium bicarbonate, and agitated with amylic alcohol to dissolve the alkaloids. This process gives a fairly constant product, but in order to insure the absence of interfering substances in some cases the sample was diluted with water and treated with lead acetate to precipitate various substances, the alkaloids being subsequently extracted from the filtrate after removing the excess of lead.

---

* It is possible that psychotrine is a decomposition product. It is known that ipecacuanha extracts differ much in the proportion of alkaloid insoluble in ether.

† The solubility of cephaëline in caustic alkalies suggests the presence in its molecule of a hydroxyl-group, which is known to exist in the morphine molecule.

The colour-tests were made in each case by taking up the alkaloidal solution in a pipette and allowing it to fall, drop by drop, on to the concave side of a cover of a porcelain crucible placed on a flask full of boiling water. To the spot of alkaloidal residue thus obtained a drop of reagent was added by means of a glass rod, and the mixture cautiously stirred. Under these circumstances the alkaloids from extract of ipecacuanha gave, when treated with ferric chloride, a blue coloration changing to green, as against a greenish blue from a residue of opium alkaloids obtained in the same manner. Fröhde's reagent gave colours varying from bluish purple to violet. The colour closely resembled that given by mixed opium alkaloids, but was not so bright as that yielded by pure morphine. Starch and iodic acid gave with some extracts of ipecacuanha an immediate blue coloration, but a negative or tardy result was obtained in other cases. Opium alkaloids, of course, gave an immediate blue. All preparations of opium and ipecacuanha immediately reduced a mixture of ferric chloride and potassium ferricyanide with production of Prussian blue.

The isolated and purified alkaloids of ipecacuanha in some instances gave reactions less striking than those obtained with the mixed alkaloidal residue. Psychotrine appears to be the constituent of ipecacuanha alkaloids which gives colour-reactions with ferric chloride and iodic acid, but we believe this to have been due, in part at least, to the presence of another body which could be separated by acetate of lead. At any rate, by treating the extract of ipecacuanha with lead acetate, and decomposing the precipitate with sulphuretted hydrogen in the usual way, we obtained a substance which was removed by amylic alcohol from acidulated solutions, and yielded an extract which immediately reduced iodic acid and mixed ferric chloride and ferricyanide. On rendering the acidulated liquid left from the amylic alcohol treatment alkaline with sodium bicarbonate, and again shaking with amylic alcohol, a substance was obtained which gave a bluish-green coloration with ferric chloride, a dirty purple with Fröhde's reagent, and an immediate blue with mixed ferric chloride and ferricyanide. These reactions appear to point to the presence of a body apparently alkaloidal in nature, which is precipitated by lead acetate.

An objection which applies to many of the colour reactions of alkaloids is that the colours obtained are really due to reduction-products of the reagents employed. Thus, for instance, almost any reducing agent will produce Prussian blue from a mixture of ferric chloride and potassium ferricyanide. If the substance has a somewhat strong reducing power, it will liberate iodine from iodic acid, and thus will give a blue coloration with starch. Similarly, the various shades of green, blue, and purple, obtained with Fröhde's reagent, are doubtless due to the formation of lower oxides of molybdenum, though the actual colours are sometimes curiously constant for the same alkaloid.

Quite recently there has been published in the *Archiv. der Pharmacie* (September 10, 1902), by G. Frerichs and N. de Fuentis Tapis, a paper on "The Assay of Ipecacuanha." These observers generally confirm the observations of Paul and Cownley. The authors state that both emetine and cephaëline dissolve in Fröhde's reagent almost without coloration, but that the addition of a trace of sodium chloride or hydrochloric acid immediately produces an intense indigo-blue coloration in the case of cephaëline, but not with emetine. Frerichs and Tapis appear to have actually dissolved the alkaloids in excess of Fröhde's reagent, which is a very objectionable method, and one which results in the destruction of all delicacy of the reaction.

According to our experience, when a drop of Fröhde's reagent is applied to an alkaloidal residue on porcelain, emetine gives, as above stated, a dirty-green colour, but this is changed by addition of a minute quantity of hydrochloric acid to a fine grass-green. Cephaëline gives a purple coloration instantly, changed by the addition of hydrochloric acid to a magnificent Prussian blue. Psychotrine gives a dull purple with Fröhde's reagent, changed by hydrochloric acid to a pale green. Opium alkaloids, when similarly treated, give the characteristic purple on addition of Fröhde's reagent, but this colour fades on the addition of hydrochloric acid. The mixed alkaloids from ipecacuanha give the intense blue reaction of cephaëline with great distinctness on addition of hydrochloric acid. This is a readily applied and highly characteristic colour reaction for mixed ipecacuanha alkaloids, and one which distinguishes them quite sharply from opium alkaloids.

Of course, extraction of the recently precipitated ipecacuanha alkaloids with chloroform affords a method of separating them from morphine when there is sufficient material to work on.

COLOUR-REACTIONS OF THE ALKALOIDAL RESIDUES FROM AMYLIC ALCOHOL EXTRACTIONS.

Reagent.	Ipecacuanha Extract, No. 1.	Ipecacuanha Extract, No. 2.		Ipecacuanha Extract, No. 3.		Opium Alkaloids.
	Direct Process.	Direct Process.	Lead Acetate Process.	Direct Process.	Lead Acetate Process.	
Ferric chloride.	Blue, changing to green.	Blue, changing to green.	Indefinite.	Blue, changing to green.	Blue, changing to green.	Greenish-blue.
Fröhde's reagent.	Bluish-purple.	Purple.	Pink, changing to blue and green.	Violet-blue, changing to dirty pink.	Violet-blue, changing to dirty pink.	Purple.
Fröhde's reagent and hydrochloric acid.	Deep blue.	Deep blue.	Deep blue.	Deep blue.	Deep blue.	Purple (fading).
Starch and iodic acid.	Immediate blue.	Blue, changing to green.	Negative.	Negative.	Pink, changing to blue slowly.	Immediate blue.
Ferric chloride and potassium ferricyanide.	Immediate blue.	Immediate blue.	Immediate blue.	Immediate blue.	Immediate blue.	Immediate blue.

COLOUR-REACTIONS OF THE ISOLATED ALKALOIDS OF IPECACUANHA.

Reagent.	Emetine.	Cephaëline.	Psychotrine.
Ferric chloride.	Indefinite.	Bluish-green (B). Indefinite (C).	Pale cherry red (B). Indefinite (C).
Fröhde's reagent.	Dirty green (B). Bluish (C).	Pink, changing to green (B). Reddish-purple (C).	Pale pink (B). Dull purple (C).
Fröhde's reagent and hydrochloric acid.	Grass green.	Prussian blue.	Pale pink, changing to pale green.
Starch and iodic acid.	Negative.	Negative.	Blue.*
Ferric chloride and potassium ferricyanide.	Gradual blue coloration.	Almost immediate blue (B). Immediate blue (C).	Immediate blue.

The foregoing tables show the colour-reactions we have observed with the alkaloids of ipecacuanha, both mixed and separated. For comparison, we give also the reactions of the mixed alkaloids of opium, as extracted from laudanum.

In some respects the separated alkaloids from Brazilian (Rio) ipecacuanha did not give absolutely identical reactions with the parallel alkaloidal preparations from Columbian (Cartagena) ipecacuanha. Where such differences were observed, the source of the alkaloidal residue is indicated by a B or C in parenthesis.

A most valuable means of detecting ipecacuanha alkaloids consists in the production of psychotrine in a crystallized form. Paul and Cownley describe the crystals as well-defined transparent prisms of a pale lemon-yellow colour. As obtained and observed by us under the microscope, psychotrine forms very minute crystals, which appear to belong to the regular system. Many of them appear to be octohedra, and closely resemble microscopic crystals of arsenious oxide. Other crystals present a remarkable resemblance to granules of rice-starch. Crystals of psychotrine for microscopic observation are readily obtained by shaking out an amylic alcohol or chloroform solution of the alkaloid with a little dilute acetic acid. The acid liquid is separated, concentrated if necessary, and placed in a watchglass, or, preferably, on a microscope-slide furnished with a cell. A watch-glass or small beaker is then moistened internally with ammonia, and inverted over the alkaloidal acetate solution. After a time the vapours of ammonia are absorbed, and liberate the alkaloid in characteristic crystals, which are observed under the microscope. There is no occasion to employ pure psychotrine for the purpose, the crystals being readily obtainable from the mixed alkaloids of ipecacuanha.

We desire to express our obligation to Mr. John Evans for his assistance in preparing various specimens of the alkaloids of ipecacuanha in a state of purity.

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* The blue coloration with starch and iodic acid was much more strongly marked in the case of the psychotrine isolated from Brazilian root than with that from Columbian root.

THE ANALYSIS OF PREPARATIONS CONTAINING OPIUM.

BY ALFRED H. ALLEN AND G. E. SCOTT-SMITH.

(*Read at the Meeting, November* 5, 1902.)

OUR attention has been frequently called to the analysis of medicines containing certain preparations of opium, especially paregoric and cough mixtures of various kinds. The analysis of such complex mixtures is by no means simple, and we do not claim to have arrived at a complete method of examination. Paregoric elixir, or compound tincture of camphor of the British Pharmacopœia, is a typical preparation of the kind. It was formerly directed to be prepared from 40 grains each of opium and benzoic acid, 30 grains of camphor, and 30 minims of oil of anise, the whole being diluted with proof spirit to 20 fluid ounces. In the last edition of the British Pharmacopœia (1898) the opium is replaced by an equivalent amount of tincture of opium, the composition of the preparation remaining otherwise unchanged. The composition of paregoric of commerce varies considerably. The spirit being the most costly ingredient offers an inducement to reduce the proportion present. This practice is objectionable, since the prescribed proportion of oil of anise cannot be kept in solution in a weak spirit. Sometimes only traces of oil of anise are present, in which case the paregoric remains clear when diluted with three or four times its volume of water. The benzoic acid is sometimes deficient in quantity, and is occasionally wholly absent, even in the case of tinctures purchased from registered pharmacists. The opium is the most important remedial constituent of paregoric elixir, and is apt to be deficient in amount or quality, besides being frequently wholly omitted. The last practice is, of course, due to the fact that preparations of opium cannot be legally sold except by registered pharmacists, and hence a preparation destitute of opium is largely substituted by general shopkeepers for the genuine paregoric or compound tincture of camphor of the Pharmacopœia. So-called "paregoric substitutes" and "paregoric without opium" are extensively prepared and sold by shopkeepers, and are even vended by costermongers in the streets of London. Some of these preparations are made simply by omitting the opium from ordinary paregoric. In one instance within our experience the opium of paregoric elixir was replaced by hyoscyamus. Potassium and ammonium bromides are also used in factitious paregoric.

If a measured quantity (25 c.c.) of paregoric be rendered distinctly alkaline with caustic soda, and evaporated to about 10 c.c., the alcohol and a portion of the camphor and oil of anise will be volatilized, and the amount of alcohol can be deduced with sufficient accuracy from the specific gravity of the distillate. On shaking the residual liquid with ether, the remaining camphor and oil of anise will be extracted. If the ether be separated, and the aqueous liquid acidulated with hydrochloric acid, benzoic acid will in some cases be precipitated; but whether it separates or remains in solution it can be dissolved out by agitating the acidified liquid with ether. On allowing the separated ethereal solution to evaporate spontaneously in a small beaker, the benzoic acid is obtained in a state fit to weigh; but a better and more rapid plan is to repeatedly agitate the ethereal liquid with water until the washings no longer redden litmus, add a little more water and a few drops of phenolphthalein solution, and titrate the liquid with $\frac{N}{20}$ caustic alkali (preferably baryta-water), which should be added until the aqueous layer acquires a pink colour, not destroyed by agitation with the ether. Each 1 c.c. of $\frac{N}{20}$ alkali required represents 0·0061 gramme of benzoic acid. If 25 c.c. of the tincture has been employed, the number of milligrammes of benzoic acid found, multiplied by 0·35, gives the grains of benzoic acid per pint of the tincture. The meconic acid extracted, together with the benzoic acid, is too small in quantity to affect the result, but its presence may be detected and the amount roughly determined by separating the ethereal layer after the titration is complete, and destroying the pink colour of the aqueous liquid by a drop of dilute hydrochloric acid. On now adding a drop of ferric chloride solution the deep purplish-red coloration characteristic of meconic acid will be produced. Meconic acid is, however, extracted with difficulty and imperfectly by agitating its acidulated aqueous solution with ether. Amylic alcohol is a far better solvent.

The detection of meconic acid in the above manner of course proves the presence of opium in the tincture. When this information alone is sought the paregoric may be diluted in a test-tube with proof-spirit till it is of a light yellow colour, and a drop or two of solution of ferric chloride then added. If opium be present, a more or less deep red coloration will be produced, owing to the formation of iron meconate. By comparing the depth of red colour with that given by a standard tincture a rough indication of the proportion of opium present can be obtained; but the amount of meconic acid in opium is too variable to allow of much stress being placed on the result obtained. It sometimes happens that paregoric is coloured with cochineal or contains a variety of tannin, in which case the coloration with ferric chloride becomes obscured. On cautiously adding hydrochloric acid, drop by drop, the colour produced by iron tannate is destroyed, while that due to the meconate persists till considerably more acid has been added.

The proportion of opium in paregoric is too small to allow of the ordinary method of determining morphine being conveniently used; but fair results, sufficiently accurate for most purposes, may be obtained by volumetric or colorimetric application of the reaction with iodic acid.

In addition to paregoric and its constituents, cough mixtures often contain other ingredients, among which may be mentioned oil of peppermint, squills, senega, horehound, and preparations of ipecacuanha. Oil of peppermint is readily recognised by the taste and odour. Squills are not readily recognised, the most hopeful plan being based upon the isolation and recognition of quillain. Senega is remarkable for the magnificent purple colour which it yields with strong sulphuric acid. The principle which gives the coloration is not extracted by amylic alcohol from either acid or alkaline solutions.

We are indebted to Mr. A. R. Tankard for valuable experiments on these and other points.

Ipecacuanha may be said to be almost a normal constituent of cough mixtures, and is of interest on account of the close resemblance of some of the colour-reactions of its alkaloids to those yielded by opium alkaloids.

A remarkable case in this connection came within our experience a few months since. A cough mixture was sold which was expected to contain opium both as laudanum and as paregoric. The analyst to whom it was submitted reported it to contain morphine in a proportion of 0·12 per cent., or more than ½ grain per ounce. A small portion of the same sample came into our hands, and we commenced its examination in the full expectation of confirming this result. Hence we submitted the mixture to a process adapted for the separation of morphine. For this purpose the sample was diluted with water, acidulated with hydrochloric acid, and the acid liquid treated with ether to remove the essential oils and any glucosides present. The acid liquid separated from the ether was then shaken with amylic alcohol, and that again separated in its turn. It was next neutralized with sodium bicarbonate and shaken with hot amylic alcohol to insure the extraction of any morphine present. The solution thus obtained was washed with water, and small portions evaporated for the application of colour-tests. The alkaloidal residue from the amylic alcohol gave only a doubtful reaction with ferric chloride,* but a very well-developed morphine reaction with Fröhde's reagent (sulphomolybdic acid), and an immediate reduction with iodic acid and a mixture of ferric chloride and ferricyanide of potassium. On the other hand, we could obtain no microscopic crystals of morphine, as may easily be obtained from such preparations containing morphine, by shaking out the amylic alcohol solution with a little dilute acetic acid, placing a few drops of the acetate solution in a watch-glass, or on a celled microscope-slide, and covering it with a watch-glass moistened with strong ammonia, so that the vapour may act on the liquid in the lower glass. After allowing it to stand for half an hour, the liquid is examined under the microscope, when the elongated prisms characteristic of morphine are readily detected. In this case, however, we failed wholly to obtain the long prismatic crystals characteristic of morphine, but obtained crystals of an entirely different kind, some of which were octahedra, closely resembling arsenious oxide, whilst others suggested the appearance of rice-starch.

At this stage our attention was called to the fact that, at an examination in analytical chemistry held last January, the candidates were required to analyse a liquid extract of ipecacuanha. Of the six who examined the extract several reported it to contain morphine. We have personally questioned three of these gentlemen, and find that they estimated the alkaloid by extracting it from an alkaline solution with amylic alcohol, and reported the presence of morphine as the result of several more or less satisfactory colour reactions. One of the candidates has stated in evidence the facts relating to this examination. Of course, by suitable means the ipecacuanha alkaloids could have been differentiated and separated from any morphine present, but the point is that, when a process suitable for isolation and determination of morphine was used, an alkaloidal residue was obtained which simulated morphine so as to be mistaken for that alkaloid. We consequently prepared the mixed alkaloids from several samples of the liquid extract of ipecacuanha by the same method which we had employed for examining the cough mixture in question. We found the alkaloidal residue to give a well-marked greenish-blue coloration with neutral ferric chloride; a crimson changing to purple and blue colour with Fröhde's reagent; an immediate blue coloration with iodic acid and starch (with certain samples, but not with all); and an immediate formation of Prussian blue from a mixture of ferric chloride and potassium ferricyanide. The microscopic crystals obtained were, however, exactly similar to those which had been prepared from the cough mixture, and in no way resembled morphine. The crystalline appearance was so characteristic that we had no hesitation in reporting that the cough mixture contained ipecacuanha, and, in view of the colour reactions which extract of ipecacuanha of undoubted purity had given, we could find no evidence of the presence of morphine. We had searched for meconic acid, with wholly negative results, by the method which we had found serviceable in other cases—namely, extraction of the acidified liquid with amylic alcohol, evaporation of the solution, and testing the residue with ferric chloride.

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* This result we attribute to the fact that we worked on a very limited amount of material. Both opium and ipecacuanha alkaloids give the well-known coloration, but the reaction is not nearly so delicate as that with Fröhde's reagent.

After reporting the presence of ipecacuanha, the person who compounded the cough mixture informed us that he had actually introduced ipecacuanha wine into the cough mixture in question, but had not made any addition of a preparation of opium. It appeared evident, therefore, that the analyst who originally examined the mixture had mistaken the ipecacuanha alkaloids present for morphine.

The case affords an interesting example of the danger of relying on colour-indications for alkaloids without a definite assurance that no interfering body is present. The remarkable manner in which the alkaloids of ipecacuanha similate those of opium in some of their reactions is not generally known, and we have felt it to be of such practical importance as to warrant us in bringing the matter before the Society in the detailed manner we have done.

DISCUSSION.

Mr. WILLIAM CHATTAWAY stated that Mr. Allen had afforded him the opportunity of examining a portion of the original cough mixture, the composition of which had been such a vexed question, and he was able to confirm the authors' statements in every respect. The cough mixture gave several of the colour-reactions of morphine, but Fröhde's reagent, followed by hydrochloric acid, gave the magnificent Prussian blue coloration which had recently been found so characteristic of cephaëline. He also confirmed the authors' statements with respect to the characteristic crystals from ipecacuanha which they attributed to psychotrine. There was no difficulty in obtaining from the sample of cough mixture in question numerous microscopic crystals of this kind, and they were quite distinct in character from crystals of morphine obtained in an analogous manner.

Mr. ALLEN said that he was glad to know that his experience in this matter was so fully confirmed by that of Mr. Chattaway. There was no reflection on anyone who had made a mistake in the past when these reactions were not recognised, but care in the future was obviously necessary lest a preparation containing ipecacuanha might be mistaken for one containing the alkaloids of opium, especially as these colour-tests were much less definite and striking with the mixed alkaloids of opium than with pure morphine. The remarkable reaction for cephaëline with Fröhde's reagent and hydrochloric acid was of great assistance, but it was conceivable that the reaction with Fröhde's reagent might be complicated by the presence of other substances, and he thought that the most definite and characteristic evidence of the presence of ipecacuanha was really that afforded by the production of crystals of psychotrine.

Mr. Moor mentioned that with regard to tinct. camph. co. a standard had been recently issued by the Irish Local Government Board. That standard did not render it necessary to go very deeply into the question, but simply laid down the specific gravity, the alcoholic strength (57 per cent. by volume), and the total solids, which were put at 0·33 per cent. He was inclined to think, however, that that percentage of total solids was somewhat too high, and would like to hear Mr. Allen's opinion on this point.

Mr. Allen said that he was afraid he could not, without reference, give any figure for the total solids in paregoric. It was to be borne in mind, however, that at present this preparation was directed to be made, not with opium, of which the quantity used to be 40 grains per pint, but with an equivalent quantity of tincture of opium. The solids of the paregoric would therefore consist of the soluble matter of the opium from which the tincture was prepared, assuming all the other constituents of the paregoric to be volatile; so that from a knowledge of the average amount of solid matter contained in the laudanum—which, however, was liable to some variation—it might be ascertained by calculation whether the standard was correct.

Mr. Moor said that the quantity of tincture of opium directed to be taken was 60·9 c.c. per litre, and, assuming the average solids of the tincture to be 4 per cent., or a minimum of 3 per cent., the total solids of the paregoric might fall as low as 0·18 per cent., assuming the benzoic acid and oil of anise to be entirely volatile; and, as a matter of fact, he had figures of his own showing as little as 0·2 per cent. in a number of samples which he believed to be quite genuine. He mentioned this because the standard referred to was intended to form the basis of prosecutions; and if that were insisted upon, any sample falling below 0·33 per cent. in total solids would be prosecuted on. He thought, however, that it was not quite correct to place the standard so high.

Mr. R. A. Cripps said that he could quite confirm Mr. Moor's remark that paregoric might be perfectly genuine, and yet contain not more than 0·2 per cent. of total solids. In the examination of cough mixture, it seemed to him that ipecacuanha was almost the first thing to be expected; and with the knowledge that the alkaloids of ipecacuanha were soluble in amylic alcohol, a reasonable course would seem to be to extract first in the alkaline condition with ether, which would remove the cephaëline and emetine and leave the morphine.

Mr. Allen said that psychotrine would also remain undissolved after such a process as that suggested by Mr. Cripps, and psychotrine was the alkaloid which gave the characteristic crystals he had described. Chloroform would be preferable, but he would be very shy of treating a small quantity of such a preparation as a cough mixture with ether or chloroform for the purpose of removing one class of alkaloids and leaving the other. As a matter of fact, however, his purpose had been rather to describe the way in which the matter had actually been dealt with in the past than to suggest what might be the best way of dealing with it.

Mr. CRIPPS said that, as far as his experience went, there was no practical difficulty in the treatment with ether for removing the alkaloids other than morphine.

NOTE ON HONEY.

By HENRY LEFFMANN.

SOME time ago I was consulted by a manufacturer of table accessories in regard to a sample of honey which had been reported by one of the State chemists as adulterated. The manufacturer did not have any portion of the sample which had been analysed, but brought me some samples which he believed to be from the same lot, and said that the material was from California, and was packed as received, being believed to be pure. Inquiry elicited the fact that the sample purchased by the State's agent had been found to give the following polarimetric results on the ordinary sugar-scale:

Before hydrolysis	9·0
After hydrolysis	3·0

The sample submitted to me gave results of the same import, although not exactly the same figures, namely:

Before hydrolysis	13·5
After hydrolysis	9·5

Dextrorotatory honey is usually looked upon with suspicion. When the sample remains strongly dextrorotatory after hydrolysis, the addition of commercial glucose is assumed.

It is, however, established that pure honey may be dextrorotatory. The following figures are reported on good authority as given by samples of undoubted purity:

Before hydrolysis	8·2	7·2	7·3
After hydrolysis	2·8	3·3	2·6

Polarimetric data are so technical that it is desirable to have some chemical tests for the presence of glucose as such. Most of the data that are herewith presented are principally of value as embodying the application of German methods to American samples, and, as will be seen, confirming the claims of the German chemists.

Commercial glucose is by no means the simple substance that it was supposed to be when first manufactured. It contains, in addition to dextrose. considerable dextrin and maltose, some nitrogenous matter, and a notable amount of unfermentable optically active carbohydrates of uncertain nature, among which is one called gallisin. As strong alcohol precipitates dextrin from glucose, the suggestion to test with this would naturally occur; but although honey is largely invert-sugar, it frequently contains considerable amounts of dextrinous matter that precipitates with strong ethyl-alcohol. Bechmann suggested the use of methyl alcohol, and tests I have recently made have shown the value of this method. The refined methyl alcohol, termed "Columbian spirit," is preferable. Two methods are employed. The simplest consists in mixing the honey with an equal weight of water and then adding a large volume of the alcohol. The quantities used have been 8 grammes of honey, 8 c.c. of water, and 100 c.c. of alcohol. The characteristic of glucose is to give a sticky precipitate which may become somewhat granular, and which adheres to the wall of the vessel. Pure honey gives a light flocculent precipitate, which does not adhere to the glass.

For the second test a solution of 20 grammes of the honey in 100 c.c. of water is prepared. Five c.c. of this solution are mixed with 3 c.c. of a 2 per cent. solution of barium hydroxide and 17 c.c. of methyl alcohol, and the mixture shaken with as little contact with air as possible. Pure honey gives but little precipitate, but in the presence of glucose or glucose syrup a considerable precipitate is formed.

These tests applied to the suspected honey and to samples believed to be pure, and also to mixtures of such samples with commercial glucose, gave very characteristic results. The barium method is unsatisfactory on account of the action of the air, which cannot be easily avoided. An attempt to substitute a solution of barium carbonate in water containing carbonic acid did not succeed. Apparently the direct action of the hydroxide upon the carbohydrates is needed.

It seemed probable that useful information might be obtained by comparing the condition of solutions before and after fermentation, including the polarimetric data, especially as applied in the examination of wine. The following experiments were made: Three solutions were prepared by dissolving 25 grammes of material in water made up to 250 c.c. The polarimetric reading of each was taken, and the solutions were then fermented at room temperature (over 20° C.) for six days, and again examined. A few drops of a solution of disodium hydrogen phosphate and ammonium carbonate had been added to each. The fermentation was quite active for several days. The results were as follows:

	Polarimetric Reading.		Fermented Solution.	
	Before Fermentation.	After Fermentation.	Specific Gravity.	Solids per Cent.
Pure honey	−6·0	3·0	1,003	1·6
Commercial glucose	65·0	43·7	1,014	4·2
One-half honey, one-half glucose ...	28·6	22·5	1,009	2·8

The above data are too few to form a basis for argument, but they tend to show that the fermentation method may aid in elucidating the nature of sample under dispute.

Some experiments were made with a view of determining the polarimetric character of solutions of samples in methyl alcohol. Crude methyl alcohol was used. In each case 8 grammes of the sample were treated with 100 c.c. of methyl alcohol, well mixed, and allowed to stand for more than a day. The clear liquid was then decanted and the reading taken. All samples were examined at the same temperature. The results were:

Sample.	Precipitate with Methyl Alcohol.	Polarimetric Reading.
Pure honey	Slight, flocculent	1·0
Suspected honey	Marked, granular	22·0
Pure honey (glucose equal parts) ...	Marked, very sticky	31·0
Glucose	Marked, sticky	10·5

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

The Detection of Added Water in Milk by Means of the Nitrate Test. M. Siegfeld. (*Molkerei-Ztg. Hildesheim*, 1902. xvi., 161, 162; through *Zeit. für Untersuch. der Nahr. und Genussmittel*, 1902, v., [18], 867.)—The author has compared the various reactions for detecting the presence of nitrates in milk, and finds that the formaldehyde test is as sensitive as the diphenylamine reaction. The former test should be made by allowing the milk, to which a drop of formaldehyde has been added, to run on to the surface of pure sulphuric acid. If nitrates be present a violet ring is formed at the junction of the two liquids. It is not advisable to mix the acid and milk, as milk containing no nitrates gives a reddish-violet coloration, which may be mistaken for the nitrate reaction.

(NOTE BY ABSTRACTOR.—Should the sulphuric acid contain a trace of iron, a blue ring is always obtained with milk containing formaldehyde, whether nitrates be present or absent.) W. P. S.

Method for the Estimation of Metallic Impurities in Condensed Milk. J. W. Abbott. (*Bull. Massachusetts Health Dept.*, 1900, 37; through *Zeit. für Untersuch. der Nahr. und Genussmittel*, 1902, v. [18], 866.)—The condensed milk is mixed in a china basin with a third of its weight of sulphuric acid, and subjected to an electric current of 1 to 4 ampères. After fifteen minutes' treatment the whole mass becomes thorougly charred. The incineration is then readily completed over a free flame, and the ash may be employed for the estimation (electrolytical) of the metallic impurities which may be present. W. P. S.

Determination of the Casein precipitated by Rennet. L. Lindet. (*Ann. de Chim. anal.*, 1902, vii., 361-363.)—According to the author's experience, the methods of determining casein in milk are not reliable, for the precipitation is invariably incomplete; and there is therefore no exact method of determining the value of milk for the purpose of cheese manufacture. The quantity of casein precipitated by rennet under definite conditions can, however, be rapidly calculated from the depression in the specific gravity of the separated milk after the addition of the enzyme. From the average results of his experiments the author finds that on the average the precipitation of each 3·5 grammes of casein per litre lowers the specific gravity of the whey by 1 degree. Hence the difference between the specific gravity of the separated milk and of the whey multiplied by 3·5 gives the amount of casein per litre precipitated by the rennet.

Instead of separating the fat from the milk the specific gravity of the fat-free milk can be calculated by means of the formula—

$$100\,D = a \times 940 + (100 - a)D',$$

$$\text{or } D' = \frac{100\,D - a \times 940}{100 - a},$$

where D represents the specific gravity of the whole milk expressed in grammes, D′ that of the separated milk, 940 that of the butter-fat in the milk, and a the percentage of butter-fat. C. A. M.

Separation and Estimation of Cholesterol in Butter Fat. A. Kirsten. (*Zeit. für Untersuch. der Nahr. und Genussmittel,* 1902, v. [18], 849-856.)—Ten grammes of the melted fat are heated on a boiling-water bath under a reflux condenser for fifteen minutes with 20 c.c. of alcoholic potash, made as follows: 2 volumes of concentrated potassium hydroxide solution—1,000 grammes per litre—1 volume of water, and 7 volumes of absolute alcohol. Forty c.c. of water are then added, and, after cooling, 50 c.c. of ether. The contents of the flask in which the saponification has taken place are transferred to a separating funnel, and the flask is rinsed out with 50 c.c. of ether several times. The funnel is then shaken vigorously for one minute, and allowed to stand, the contents quickly separate. The ethereal layer is removed, and the soap solution shaken out five times with ether, using 50 c.c. of the latter each time. Should an emulsion form, a little alcohol may be added. The ether is distilled off from the combined extracts, and the alcohol remaining in the residue is removed by evaporation on the water-bath. The residue is then again heated with 10 c.c. of alcoholic potash, to saponify traces of fat which it still contains, diluted with 30 c.c. of water, and shaken out with 100 c.c. of ether. After drawing off the soap solution, the ethereal layer is washed three times with 10 c.c. of 5 per cent. potassium hydroxide solution and twice with 10 c.c. of water. These washings are not added to the soap solution. The ether is filtered into a weighed flask. After once more shaking the soap solution with 100 c.c. of ether, this second ethereal portion is also filtered into the weighed flask, and the filter washed with a little ether. The ether is then distilled off, and the residue of cholesterol is dried and weighed. The average amount of cholesterol found by the author in butter fat was about 0·46 per cent. At the commencement of lactation the quantity was 0·37 per cent., increasing at the end of the lactation period to 0·50 per cent. W. P. S.

Detection of "Vinegar Essence" in Fermentation Vinegar. F. Rothenbach. (*Deutsche Essigindustrie*, 1903, vi., 49, 50 and 59-64; through *Zeit. für Untersuch. der Nahr. und Genussmittel*, 1902, v., [17], 817-819.)—The following methods are given:

1. Fifty c.c. of the vinegar are shaken in a separating-funnel with 20 to 30 c.c. of pure, alcohol-free chloroform. An emulsion is avoided by adding water and well cooling the mixture. The chloroform layer is filtered through a dry filter and again cooled, when a turbidity forms. On adding 2 to 3 c.c. of a mixture of 10 parts of concentrated sulphuric acid and 11 parts of fuming nitric acid, a dark-red zone appears between the two liquids should fermentation vinegar be present. By carefully shaking the mixture the whole chloroform layer becomes red-coloured, the acid layer remaining colourless. The red colour is very stable. Vinegar essence gives no coloured zone.

2. One c.c. of the sample, 0·1 c.c. of $\frac{N}{10}$ iodine solution, and 0·2 c.c. of concentrated sulphuric acid are mixed in a test-tube and cooled. Concentrated vinegar essence gives a clear, dark-red coloration, and water containing 1 per cent. of essence a clear yellow colour. Pure fermentation vinegar yields a dark-red coloration; the fluid soon becomes turbid and opaque, and a greenish layer forms on the surface. Dilute fermentation vinegar gives a dark-red colour, soon becoming turbid. Mixtures of 100 c.c. of 10 per cent. fermentation vinegar with 20, 10, and 5 c.c. of vinegar essence give clear red colours; with less than 2·5 c.c. the mixture is still red, but soon becomes turbid. When fermentation vinegar is distilled the distillate yields a clear, bright-yellow colour, whilst the residue gives a turbid red coloration. Vinegar essence which has been allowed to stand in contact with vinegar shavings for a considerable time shows, when tested by this method, a dark-red colour which remains clear.

3. The vinegar to be tested is diluted down to 1 per cent. Twenty c.c. of this dilute solution are placed in a wash-bottle, which is connected to a second wash-bottle containing 5 c.c. of sulphuric acid (1 : 3) and 19 c.c. of $\frac{N}{100}$ permanganate solution. A strong regular current of air, which has first been passed through an alkaline solution of permanganate, is drawn through the vinegar and permanganate solutions for two minutes. The wash-bottle containing the latter is then detached and placed for five minutes on a boiling-water bath. Ten c.c. of $\frac{N}{100}$ oxalic acid solution are then added, and the titration completed with $\frac{N}{100}$ permanganate. A blank test should be made, using 20 c.c of water. Vinegar essence destroys practically no permanganate. Fermentation vinegar, according to the amount of alcohol, etc., it contains, uses at least 1 c.c. of the permanganate. An attempt is made to render this test quantitative, but without satisfactory results. W. P. S.

The Estimation of Caffeine. J. Katz. (*Zeits. f. angew. Chem.*, 1902, xxxix., 1013.)—The method is a modification of that of Beitter. Of the sample, 10 grammes are taken and shaken for half an hour with 200 grammes of chloroform and 0·5 gramme of ammonia. After settling, the chloroform solution is filtered through a Sander filter in order to obtain 150 grammes of clear water-free filtrate. The chloroform is distilled off entirely, and the residue is dissolved in 5 c.c. of ether, then 20 c.c. of 0·5 per cent. hydrochloric acid are added, and the ether is boiled away. When cold, the aqueous liquid is filtered, the flask and filter being washed several times with 0·5 per cent. hydrochloric acid, and the acid filtrate is shaken out four times with chloroform, using 20 c.c. each time. The chloroform solution is filtered if necessary, and the solvent is distilled off.

The above method is used for coffee-beans, black tea, guarana, and kola-nuts. In the case of Paraguay tea, the material is treated with chloroform and ammonia as already described, the solution evaporated, and the residue dissolved in ether. Water is then added, the ether is boiled away, and 2 c.c. of water are introduced, having 5 per cent. of lead hydroxide in suspension. The liquid is heated for ten minutes on the water-bath, then a few decigrammes of calcined magnesia are added, and the liquid after cooling is filtered. The filtrate, which should be clear and only slightly coloured, is extracted in a "perforator" for two hours or shaken out with chloroform, and the extract is evaporated down. A. M.

A Test for Phenacetin. F. H. Alcock and W. Wilkins. (*Pharm. Journ.*, 1902, 258.)—A purple coloration is obtained on strongly heating 0·01 gramme of phenacetin with 5 c.c. of pure sulphuric acid. On pouring the solution, when nearly cold, into a large bulk of water and adding ammonium hydroxide, a deep purple-coloured solution results. Should any charring have taken place, the solution must be filtered before making ammoniacal. Phenazone, sulphonal, and acetanilide give entirely different results. W. P. S.

TOXICOLOGICAL ANALYSIS.

The Forensic Value of "Florence's Crystals." N. Bocarius. (*Vierteljahrschr. gericht. Med. u. öffentl. Sanitätsw.*, 1901, xxi., 255-266; through *Zeit. für Untersuch. der Nahr. und Genussmittel*, 1902, v., 942, 943.)—Florence's crystals (see ANALYST, xxiii., 320) are not only obtained with human seminal fluid, but also with that of animals as well as with other animal and vegetable substances, such as extracts from the liver, the blood of common insects, caterpillars, etc. The crystals are always obtained even from the smallest trace of human semen, whether it be wet or dry, fresh or decomposed, except when the semen is coloured emerald-green or orange by micro-organisms. Animal seminal fluid and bodies not of a spermatic nature react more feebly with the iodine (Florence's) solution than does human semen.

The crystals may be obtained with various iodine solutions, but the reagent must contain excess of iodine. The age of the seminal traces does not influence the reaction, nor does a high temperature or decomposition, but the presence of large quantities of blood, and other animal fluids, interferes with the formation of the crystals; an excess of liquid—water or reagent—as well as an excess of potassium iodide in the latter, should be avoided. The form of the crystals is altered according to the reagent used, and by other causes. The actual substance which forms the crystals in the presence of iodine exists not only in seminal fluids, but also in many organic bodies. Florence's reagent is, therefore, only a selective test. W. P. S.

The Nature of the Substance which causes the Formation of "Florence's Crystals." N. Bocarius. (*Zeit. physiol. Chem.*, 1902, xxxiv., 339-346; through *Zeit. für Untersuch. der Nahr. und Genussmittel*, 1902, v., 943.)—To ascertain the nature of these crystals the author carried out a number of experiments with the seminal fluids of men and horses, the livers of oxen, and human brains and livers. These were first treated with 2 per cent. formaldehyde solution, and by acting on the extracts of the various objects with a solution of iodine in sodium iodide large quantities of crystals were obtained. The latter keep well if allowed to stand in the iodine solution. After decomposing the crystals with silver hydroxide, the products were treated with alcohol, and converted into platinum chloride salts. On analysis the latter were found to the following percentages of platinum: human semen, 31·65; animal semen, 31·51; human liver, 31·82; ox liver, 31·53; and human brain, 31·61. The average of these figures is 31·62 per cent., whilst choline-platinum chloride gives 31·64, and semen-platinum chloride 38·21 per cent. of platinum. The author comes to the conclusion that choline must be considered as the substance which forms the crystals with the iodine solution. (See ANALYST, xxiii., 320.) W. P. S.

Determination of Mercury in Toxicological Work. C. Pierpaoli. (*Boll. Chim. Farm.*, 1902, xli., 561; through *Chem. Zeit. Rep.*, 1902, 265.)—According to the author, the partial loss of mercury which occurs when animal substances have been destroyed by the Fresenius-Babo process is due to the following causes: In the precipitate produced by sulphuretted hydrogen the mercuric sulphide is accompanied by organic matter containing chlorine, from which it cannot be freed by washing with water; on the contrary, by excessive washing part of the sulphide passes into the colloidal modification, and is therefore lost. When the dry impure sulphide comes to be treated with nitric, and afterwards with sulphuric, acid, in order to complete the destruction of organic matter, part of the mercury is converted into chloride by the chlorine present, and volatilized when the sulphuric acid is warmed. As a matter of fact, Pierpaoli has found that if mercuric chloride is heated with sulphuric acid to a temperature not exceeding 170° C. the salt is not decomposed, but volatilizes unchanged. F. H. L.

ORGANIC ANALYSIS.

The Composition of Human Fat. H. Jaeckle. (*Zeit. physiol. Chem.*, 1902, xxxvi., 51; *Chem. Rev. Fett-u. Harz-Ind.*, 1902, ix., 232.)—According to the results of the author's analyses, normal adult human fat consists in the main of glycerides of oleic, palmitic, and stearic acids, with traces of lower fatty acids. The fat of a child during the first few months shows pronounced differences from the adult fat, being characterized by a high proportion of lower fatty acids and a small proportion of oleic acid. It was not found possible to trace any influence of the food of different individuals upon the composition of the fat. In pathological cases the fat may undergo very great changes (*cf.* ANALYST, xxi., 171). C. A. M.

The Molecular Equivalent of Insoluble Fatty Acids. M. Tortelli and A. Pergami. (*Chem. Rev. Fett-u. Harz-Ind.*, 1902, ix., 182-184, 204, 205.)—The ordinary process of determining the molecular equivalent of fatty acids consists of dissolving a weighed quantity in alcohol and titrating the solution with $\frac{N}{10}$ alkali. It has usually been assumed that the acid value thus found is identical with the saponification value. The authors, however, point out that there is a great discrepancy between the recorded molecular equivalents of the fatty acids of different oils and their known composition. The cause of this source of error, which is clearly shown in the following examples, is to be attributed to the presence of lactones :

Oil or Fat.	Oil or Fat.		Insoluble Fatty Acids.		Mean Molecular Equivalent of Fatty Acids.		Difference.
	Acid Value. I.	Saponification Value. II.	Acid Value. III.	Saponification Value. IV.	Calculated from Acid Value. V.	Calculated from Saponification Value. VI.	V.-VI.
Almond oil :							
2½ years old	1·50	193·6	196·0	202·2	286·2	277·5	8·7
Fresh, cold-drawn ...	0·70	193·6	195·8	203·2	286·5	278·3	8·2
Cotton-seed oil :							
2½ years old	0·19	195·2	194·3	204·5	288·7	274·3	14·4
Fresh, cold-drawn ...	3·80	198·3	200·9	203·1	279·2	276·2	3·0
Colza oil :							
2 years old	3·7	172·2	178·3	182·5	314·6	307·4	7·2
Fresh, cold-drawn ...	0·55	174·9	176·6	181·2	317·7	309·6	8·1
Castor oil :							
2½ years old	3·29	—	183·1	189·0	306·4	296·7	9·7
Fresh, cold-drawn ...	1·9	181·3	187·0	191·1	300·0	294·3	5·7
Linseed oil :							
3 years old	2·70	192·6	191·5	205·4	292·8	273·2	19·6
Fresh, cold-drawn ...	0·30	189·8	194·6	201·8	288·2	277·9	10·3
Ox tallow :							
2 years old	4·3	199·6	205·4	207·1	273·1	270·8	2·3
Fresh, rendered by heat	1·4	200·0	205·7	209·9	272·7	267·2	5·5
Lard :							
2 years old	0·2	196·8	203·4	203·1	275·8	276·2	0·4
Fresh, rendered by heat	5·9	196·8	205·0	204·8	273·4	273·9	0·5
Palmitic acid	—	—	202·7	218·7	276·5	256·4	20·1
Stearic acid	—	—	198·7	198·9	282·3	282·0	0·3
Oleic acid, from olive oil...	—	—	199·5	201·4	281·2	278·5	2·7

To obviate this error, the authors hydrate the lactones by boiling the fatty acids with an excess of alkali and titrate the excess, just as in the determination of the saponification value by Koettstorfer's method. C. A. M.

Some Colour Reactions of Fatty Oils. H. Kreis. (*Chem. Zeit.*, 1902, xxvi., 1014.)—This is essentially a continuation of the author's previous article (ANALYST, xxvii., 330). It is known that Bishop's reaction—a green colour on shaking with 1·19 hydrochloric acid—only occurs with such sesamé oils as have been bleached by insolation or otherwise. It is also known that, if a sesamé oil that does not give Bishop's reaction is mixed with some other insolated or bleached oil, the whole gives a strong green or blue colour when treated similarly. Moreover, a list of oils was contained in the former paper which no longer yielded the Bellier test, but which behaved with sesamé as above. For convenience this reaction may be termed the Bishop-Kreis test. It is now found that oils which yield the Bishop-Kreis reaction also give colours with resorcinol and with phloroglucinol, the test being preferably made by mixing 2 c.c. of 1·19 HCl with 2 c.c. of oil and 2 c.c. of a cold saturated solution of resorcinol in benzene, or of a solution of 1 part of phloroglucinol in 1,000 parts of ether. After a short time—or rapidly on the water-bath—fine violet or red tints appear in the acid, which are not affected by the addition of water. Hitherto this reaction has been observed with olive, arachis, sesamé, cotton, poppy, and walnut oils, also with two samples of butter which had become tallowy. A sample of arachis, which gave the Bellier test powerfully, but failed to yield the Bishop-Kreis test, was exposed to sunlight for eight (September) days; it then failed to give the Bellier, but gave a notable green with the Bishop-Kreis, and also yielded bright colours with resorcinol or phloroglucinol and HCl. The acid value (4·4) was not affected by the insolation.

F. H. L.

Detection of Sesamé Oil in Earth-Nut Oil. J. Schnell. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1902, v., 961-963.)—Soltsien's reaction is recommended, the test being carried out as follows: The oil is mixed with an equal volume of stannous chloride (German Pharmacopœia strength) and shaken vigorously, but not repeatedly. The test-tube containing the mixture is then placed in boiling water until separation takes place. A red coloration appears should sesamé oil be present, whilst pure earth-nut oil gives no coloration. Less than 1 per cent. of sesamé oil can be detected, but attention is drawn to the fact that sometimes the same press is used for sesamé seeds as for earth-nuts, and the oil from the latter may, for this reason, show indications of the presence of traces of sesamé oil.

It may be mentioned that the stannous chloride reaction is particularly applicable to butters and margarines artificially coloured with coal-tar dyes. The colours are reduced and rendered colourless when the test is applied, and their previous extraction with dilute hydrochloric acid is unnecessary.

Several samples of earth-nut oil from West Africa examined by the author gave iodine numbers as low as 84·4 to 85·7. Others, from the East Indies, gave higher numbers than usual—from 89·7 to 95·0. As these numbers are higher than that for pure olein, other unsaturated acids besides oleic are probably present in these samples. W. P. S.

Halphen's Reaction for Cotton-Seed Oil. B. Sjollema and J. E. Tulleken. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1902, v., 914-916.)—Butter obtained from cows which have been fed on cotton-seed meal gives with Halphen's test exactly the same coloration as does cotton-seed oil itself. On spectroscopically examining the colour produced in this test the authors found that, at a low temperature (55° C.), cotton-seed oil yielded an absorption band in the yellow with a maximum at λ 550, a thicker layer showing a maximum more to the left at λ 570. By long heating or by employing a higher temperature a second band was produced at λ 490, which gradually increased in intensity. A thicker layer, without dilution, caused the right half of the spectrum to become so dark that observations were rendered impossible. On considerably diluting with amyl alcohol, the band had a distinct maximum at λ 490, but the band in the yellow had lost much of its intensity. W. P. S.

Notes on Some Essential Oils. (*Schimmel and Co.'s Half-Yearly Report*, October, 1902; through *Chem. Zeit.*, 1902, xxvi., 1005.)

Camphor Oils.—Methods of manufacture having been somewhat altered, these oils are rather different from what they formerly were. The light oil has a yellowish colour and a specific gravity of about 0·900; the dark oil is green, and its density is 0·930. A third product is now prepared, viscid, and of a fine blue colour; it is used in porcelain painting. It boils between 280° and 300° C., has a specific gravity of 0·95 to 0·96 at 15° C., and is dextrorotatory, one sample showing a value of +32° 55′. Apparently it consists for the most part of an alcoholic body, for on treatment with acetic anhydride it yields a considerable saponification value. In spite of its colour, it does not tint soap, and it is therefore employed as a fixing agent for ordinary soap perfumes. Little is yet known of the constituents of camphor oil which are soluble in alkali. On distillation aldehydes and acids are obtained, but the latter have not been identified. A comparatively small quantity of eugenol is present in the higher fractions. In the highest acid fractions eugenol and carvacrol have been found; the latter boils at 86° to 88° C. at a pressure of 2 millimetres. Apparently a second product, boiling at 94° to 99° (3 millimetres), is also present. About 3 per cent. of the portion soluble in alkali dissolves in dilute sodium carbonate; it consists of a mixture of the carboxylic acids of the fatty series, caprylic acid being predominant. Through its easily soluble calcium salt a liquid acid boiling at 114° to 115° (4 millimetres) has been recovered from the crude acids; analysis of its silver compound leads to the formula $C_9H_{16}O_2$; it probably belongs to the oleic series.

Oil of Bergamot.—S. Gulli has published an account of this oil. It is only distilled between February and April, when the trees are pruned and stripped; the yield is quite small, 100 kilos of leaves giving but 150 grammes of oil; probably not more than 20 or 25 kilos are made in any year. The pure oil has a specific gravity of about 0·870 to 0·873, its rotatory power is +25° to +26°, and it is soluble in an equal volume of 90 per cent. alcohol. It contains about 32 to 34 per cent. of esters reckoned as linalyl acetate, part being the methyl ester of anthranilic acid.

Oil of Lemon.—Although a large number of bodies have been identified in this oil—*d*-limonene, cymol (?), phellandrene, citral, citronellal, geranyl acetate, a sesquiterpene, octyl- and nonyl-aldehyde, and pinene—it is not possible by artificially mixing these substances together to produce a useful lemon oil. This is due to the fact that several important odour-giving constituents have been overlooked. Recently in Schimmel's laboratory methylheptenone has been obtained from the distillate of lemon oil, and terpineol (melting at 35°, Δ'-Terpene-8-ol) recovered from the residue.

Oil of Lemon-grass.—Owing to the lack of prosperity in the sugar industry of the West Indies, it would seem that attempts are being made to cultivate the lemon-grasses in those islands. The superintendent of the botanical gardens in Trinidad has published an account of some investigations made in the Government laboratory at Antigua on this subject. The oil of *Andropogon Nardus var.* has been found to have a specific gravity of 0·9084 at $\frac{15{\cdot}5^\circ}{15{\cdot}5^\circ}$; a specific rotatory power of + 0° 1′; an aldehyde content of 15·5 per cent.; saponification value, 23; and saponification value after acetylization, 168·6, corresponding with about 53 per cent. of total alcohols. The oil of *Andropogon Schœnanthus* gave: Specific gravity, 0·9315 at $\frac{15{\cdot}5^\circ}{15{\cdot}5^\circ}$; rotatory power, +3°; aldehyde content, 48·2 per cent.; saponification value, 31·1; after acetylization, 69·6, corresponding with 20·2 per cent. of $C_{10}H_{18}O$. Apart from its slight dextrorotatory power, the former oil resembles Ceylon citronella oil, for it is not soluble in 10 volumes of 70 per cent. alcohol, but dissolves in the same volume of 80 per cent. spirit; but the latter has very different properties from Palmarosa oil, with which it appears to be botanically identical. Palmarosa oil, however, is not a lemon-grass oil, for it contains too little aldehydes, assuming, that is, that its aldehyde is really citral.

Liquid Oil of Musk.—Ordinary oil of musk is solid at ordinary temperatures, owing most probably to the presence of palmitic acid. This being inconvenient in practice, Schimmel and Co. have removed the solid constituent, and have introduced a liquid oil. The new material remains fluid at all temperatures, and is equivalent to six times its quantity of the usual product. The liquid oil has a specific gravity of 0·909, a rotatory power of +1° 10′, an acid value of 2·4, an ester number of 180·5, and it is soluble in 5 to 6 volumes of 80 per cent. alcohol.

Oil of Neroli.—A recent investigation has shown that oil of neroli contains, besides the constituents hitherto recognised, the following bodies: *l*-pinene, *l*-camphene (?), dipentene, decyl-aldehyde (?), an alcohol $C_{10}H_{18}O$ (probably *l*-linalool), phenylethyl alcohol (free, or as ester), *d*-terpineol (melting-point, 35°), phenylacetic acid, and benzoic acid.

Oil of Petit-grain.—A specimen of genuine oil from Paraguay has been found to have a specific gravity of 0·8912, and to contain furfurol, *l*-pinene (?), *l*-camphene (?), dipentene, an alcohol $C_{10}H_{18}O$ (*l*-linalool), *d*-terpineol (melting-point, 35°), geraniol, geranyl acetate, and traces of a basic substance.

Italian Oil of Peppermint.—A sample of this oil, distilled in Piedmont, had a pale greenish-yellow colour and an odour somewhat resembling pennyroyal. Its constants were: specific gravity at 15°, 0·9122; rotatory power, −16° 21′; solubility, 7 volumes of 70 per cent., 1·1 volume of 80 per cent. alcohol; total menthol content, 52·5 per cent., 7·89 per cent. thereof existing as esters. The sample, accordingly, did not solidify in a freezing mixture. The menthone, on the contrary, was high—viz., 22 per cent. With larger quantities of alcohol than those mentioned the oil gave a transient opalescence.

Oil of Aniseed.—Tardy has recently investigated this oil. After removing the bulk of the anethol, the sample had a rotatory power of −3° 15′. Besides the constituents already known—pinene, phellandrene, methylchavicol, the ethyl ester of quinol, anisaldehyde, and anisic acid—Tardy found anisic ketone, a sesquiterpene having a lævorotatory power of 5°, and a body melting at 212°, which is probably identical with the substance melting at 213° C. isolated from oil of fennel. Tardy also considers that oil of aniseed contains terpineol, but beyond quoting the boiling-point of 216° to 218° he gives no proof of its existence.

Oil of Bystropogon Origanifolius.—This is a new material introduced by the firm of L. Rigal in Teneriffe. It is prepared from a shrub of the Labiate species which grows largely in the Canary Islands. It forms a bright yellow oil with an odour recalling that of pennyroyal. Its constants are: specific gravity at 15°, 0·9248; rotatory power, +2° 57′; acid value, 0; saponification value, 11·1; after acetylization, 58·83; coefficient of refraction, 1·48229; solubility, 2·5 volumes of 70 per cent., 0·7 volume of 80 per cent. alcohol. The oil distils mainly between 162° and 234° C. Its chief constituents are pulegone and menthone; it contains but little limonene.

F. H. L.

Detection of Carbazol and Phenanthrene in Anthracene. H. Behrens. (*Rec. trav. chim. des Pays-Bas et de la Belge*, 1902, xxi., 252; through *Chem. Zeit. Rep.*, 1902, 266.)—To detect carbazol, anthracene is extracted with cold acetic ether and the liquid evaporated to dryness. The residue is again taken up in a few drops of the same solvent, and allowed to evaporate on a watch-glass. The carbazol separates out at the edges, the anthracene in the middle of the mass. A little of the former portion is removed with a needle, and treated with a small drop of nitrobenzene in which a few little crystals of phenanthrene-quinone have been suspended. In the presence of only 0·5 per cent. of carbazol copper-coloured crystalline plates are produced, pure anthracene giving no reaction. To detect phenanthrene, the anthracene is extracted with benzene and the solid portion of the extract treated with nitrobenzene containing some *a*-dinitrophenanthrene-quinone. If the original anthracene contained only 1 per cent. of phenanthrene, the existence of the latter will be shown by the formation of the characteristic brown rod-like crystals of the condensation product.

F. H. L.

The Extraction of Alkaloids from Alkaline Solutions. E. Springer. (*Pharm. Zeit.*, 1902, xlvii., 82, 83; through *Zeit. für Untersuch. der Nahr. und Genussmittel*, 1902, v., 935, 936.)—By employing an apparatus in which aqueous solutions may be extracted continuously with immiscible solvents (see ANALYST, 1892, p. 44), it was found that the whole of the commonly occurring alkaloids can be quantitatively extracted with chloroform in this manner. Morphine, not being readily soluble in pure chloroform, is best extracted with chloroform containing 10 per cent. of alcohol, after first liberating the morphine by ammonia or sodium hydrogen carbonate, as sodium hydroxide is not suitable. In the case of veratrine, codeine or cocaine, an excess of alkali is to be avoided. Sodium hydrogen carbonate should be employed to liberate codeine from its salts, as sodium hydroxide is liable to cause the retention of small traces of the alkaloid in the aqueous solution. Strychnine is rapidly extracted from solutions containing sodium hydroxide, and in traces even from acid solutions. Cocaine is also readily extracted, but it should be noted that it is liable to decomposition at moderately low temperature; chloroform extracts traces of this alkaloid from acid solutions. Traces of atropine and narcotine are also obtained from acid solutions, whilst the extraction is rapid when the solution is alkaline. Coniine and nicotine must be extracted at quite low temperatures. Other solvents, such as isobutyl alcohol, amyl alcohol, benzene, toluene, carbon tetrachloride, and petroleum may, in certain cases, be used instead of chloroform. W. P. S.

The Extraction of Alkaloids from Acid Solutions and Salts of the Alkaloids from Aqueous Solutions. E. Springer. (*Apoth. Zeit.*, 1902, xvii., 225, 226.)—Chloroform was found to extract alkaloids most readily from solutions rendered acid with hydrochloric acid as compared with other acid solutions, and it follows that the hydrochlorides are comparatively more soluble than other salts of the alkaloids. In testing for traces of alkaloids hydrochloric acid solutions should not be shaken out with chloroform, unless these extracts are tested for alkaloids as well as the alkaline extractions. From sulphuric acid and phosphoric acid solutions less alkaloid is removed by chloroform, and in the case of solutions acidified with tartaric acid very little. Only aconitine and narcotine salts are appreciably soluble in chloroform. Codeine, cocaine, quinine, morphine, coniine, and nicotine salts are practically insoluble, and strychnine, atropine, and veratrine only yield a minute trace of soluble salt. Morphine is not dissolved by pure chloroform from acid solutions, but is to some extent soluble in chloroform containing 10 per cent. of alcohol.

Morphine hydrochloride, morphine sulphate, and quinine sulphate are not dissolved by chloroform from neutral solutions; cocaine hydrochloride gives traces of the free base. All the other alkaloids are extracted from neutral solutions, the soluble portion consisting partly of the salt and partly of free base. The more concentrated the solutions are, the more is dissolved by the chloroform. The presence of glycerol, saponin, etc., has no influence on the solubilities. W. P. S.

Detection of Blood Colouring-Matters in Urine. O. Rossel. (*Schweiz. Wochschr. Chem. Pharm.*, 1901, xxxix., 557, 558; through *Zeit. für Untersuch. der Nahr. und Genussmittel*, 1902, v., 942.)—The urine is strongly acidified with acetic acid and shaken out with an equal volume of ether. A drop of water is added to the ethereal extract, and then 15 to 30 drops of old oil of turpentine or 5 to 6 drops of fresh hydrogen peroxide. After shaking, 10 to 20 drops of a freshly-prepared 2 per cent. solution of Barbadoes aloïn in alcohol (70-90 per cent.) are added, and the mixture is again well shaken. In the presence of minute traces of blood—too small to be detected spectroscopically—the aqueous layer becomes distinctly red in colour within three minutes. After ten minutes the colour turns to bright cherry red.

W. P. S.

A New Colorimetric Method of determining Mercury in Urine. Schumacher and W. Jung. (*Zeit. anal. Chem.*, 1902, xli., 461-484.)—As a rule, 500 c.c. of the urine are taken, but if subsequently no coloration is given with hydrogen sulphide, the amount of mercury, if present, cannot exceed 0·6 milligramme per litre; and in such cases a litre or more of the urine should be concentrated on the water-bath, with the addition of a few grammes of sodium chloride to prevent any mercuric chloride volatilizing. The 500 c.c. of urine are heated to the boiling-point with 50 c.c. of concentrated hydrochloric acid and about 5 grammes of potassium chlorate, after which the contents of the flask are cooled to about 80° C., and treated with 12 grammes of pure zinc filings. Eventually an additional 3 grammes of zinc are added, and the flask set aside for two hours. The supernatant liquid is then decanted from the deposit of zinc particles (which will have combined with all the mercury present), and this deposit washed twice with water, then treated for a few minutes with a dilute solution of sodium hydroxide, and again washed twice with water. The residue is now mixed with 50 c.c. of dilute hydrochloric acid and a little potassium chlorate, and heated gently over a small flame until everything has dissolved, more potassium chlorate being added from time to time. The solution is cooled to 70° to 80° C., mixed with about 5 c.c. of alcohol, again boiled, cooled, and transferred to a 100 c.c. flask. The liquid is mixed with a few c.c. of hydrogen sulphide water and made up to the mark, and the colour compared with that given by a freshly-prepared standard solution of mercuric chloride.

The authors state that this is the most rapid and simple method known, and they show by the results of test experiments that it is extremely accurate, whilst being capable of determining 0·1 milligramme of mercury per litre after concentrating the original urine from 3 litres to 1 litre.

C. A. M.

The Examination of Civet. A. Hébert. (*Bull. Soc. Chim.*, 1902, xxvii., 997-1000.)—The samples of guaranteed purity examined by the author melted at 36° to 37° C.

The principal constituent of civet is soluble in ether, benzene, chloroform, and petroleum spirit, but is only soluble with difficulty in alcohol, methyl alcohol, and acetone at the ordinary temperature. The residue left on treating civet with organic solvents consists of accidental impurities, hair, etc., In three samples examined by the author it ranged from 3·6 to 5·3 per cent.

The ash varied from 0·8 to 1·2 per cent., and consisted of sulphates (in large quantity), carbonates, phosphates, and other salts of iron, aluminium, calcium, magnesium, potassium, and sodium. Chlorides were almost entirely absent. The rotatory power of a filtered solution in a mixture of alcohol and ether was practically nil.

When civet is distilled in a current of steam nearly the whole of the malodorous part (skatole) passes over, whilst the residue has a musk-like odour, and resembles fat in appearance. Civet is not readily saponified, and the boiling with alcoholic alkali must be continued for a very long time. When the saponification product is taken up with warm water an opalescent solution is obtained, and on acidulating this with sulphuric acid a layer of fatty acids rises to the surface. In the three samples examined by the author these melted at 39° C., and amounted to from 51 to 70 per cent. of the original material.

The composition of civet can be considerably influenced by the method of extraction. In some cases the instrument used for the incision is smeared with honey, and the civet then reduces Fehling's solution, and has a rotatory power. Fat is also commonly used to smear the instrument, and in the author's opinion this was the case with the samples examined by him. C. A. M.

INORGANIC ANALYSIS.

Technical Estimation of Silver Bromide in Photographic Dry Plates. V. Bellach. (*Allg. Phot. Zeit.*, 1902, ix., 165; through *Chem. Zeit. Rep.*, 1902, 276.)—The film is brought into a porcelain basin and treated in the cold with a 1 : 20 solution of potassium cyanide for about fifteen minutes. When all the silver has been dissolved the liquid is rinsed into a beaker, and warmed with strong nitric acid to drive off the hydrocyanic acid. The silver bromide is thus reproduced, and coheres into a mass which can easily be washed by decantation. Finally, it is thrown on a tared double paper, washed thoroughly, dried at 110° or 120° C., and weighed. Alternatively, the cyanide solution may be electrolysed with a current of 3·7 to 4·1 volts at a density of 0·2 to 0·5 ampère per 100 square centimetres.

F. H. L.

Strong Sulphuric Acid as a Solvent in the Analysis of Tin Alloys. H. Nissenson and F. Crotogino. (*Chem. Zeit.*, 1902, xxvii., 984.)—This is essentially a continuation of the authors' former paper (ANALYST, xxvii., 336). In the absence of lead 0·5 gramme of the sample is heated with 7 c.c. of strong sulphuric acid for a few minutes till solution is complete. The liquid, which contains the tin, is cooled, cautiously diluted with hot water, and the yellow precipitate, comprising all the tin and antimony (the latter in the lower state of oxidation), is allowed to settle in a hot place. As it is, it filters better than the similar precipitate obtained after treatment with nitric acid, but it may be improved in this respect by oxidation, preferably by means of ammonium persulphate. After washing, it is strongly ignited with the filter-paper, and weighed as $SnO_2 + SbO_2$.* The filtrate is raised to the boiling-point and the copper is thrown down with thiosulphate; in the next filtrate iron, cadmium, etc., are determined. A second sample is dissolved as before, and the solution is diluted with a little water and 15 c.c. of weak hydrochloric acid. The antimony (with any copper) is precipitated with iron wire, dissolved in aqua regia, thrown down with sulphuretted hydrogen, dissolved in sodium sulphide, and the antimony itself is recovered by electrolysis. The insoluble metallic sulphides are taken up in nitric acid, and the liquid is examined for copper, bismuth, etc. If no iron and but little copper are present the antimony can be estimated by treating the original solution with hydrochloric and tartaric acids and titrating with iodine, or Györy's direct process with potassium bromate may be employed. If a volumetric method is decided upon, the sulphuric acid must be cooled immediately the alloy is dissolved, or some of the antimony may be oxidized.

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* [Ignition of a mixture of tin and antimony oxides with the filter-paper causes loss by volatilization.—B. B.]

In the presence of lead and tin only, the sulphuric acid solution is diluted with hot water, and a considerable quantity of ammonium oxalate is added. After cooling the lead sulphate is filtered off, and the filtrate is electrolysed with a current of 1 to 1·5 ampères per 100 square centimetres to recover the tin—such being the method for soft solders. If other metals are expected, the first solution is diluted with hot water only, the residue washed with weak sulphuric acid, ignited and weighed as $PbSO_4 + SnO_2 + SbO_2$. In the filtrate, copper, iron, cadmium, zinc, etc., are determined as usual. A second sample is treated identically, but the precipitate of lead, antimony, and tin is dissolved in hot dilute hydrochloric acid, two drops of sulphuric acid are introduced, the whole is cooled, and the lead is filtered off. In the filtrate antimony is thrown down with iron wire as before, and the tin either estimated by difference or by precipitation with sulphuretted hydrogen after removal of the elemental antimony. If the solution containing iron is evaporated to free it from hydrochloric acid till fumes of sulphuric acid appear, the tin will not be thrown down quantitatively by mere dilution with water, and the precipitate which is obtained will be contaminated with much iron.

The method is not suitable for the analysis of alloys (bronze and the like) containing much copper, as the dissolution does not proceed rapidly enough.

F. H. L.

Photometric Determination of Iron. J. I. D. **Hinds** and **Myrtis Louise Cullum.** (*Journ. Amer. Chem. Soc.*, xxiv., 848.)—This paper constitutes the first application of the photometric method to the case of coloured precipitates. For the readings a candle and simple photometric cylinder were used (*Journ. Amer. Chem. Soc.*, xviii., 661). The iron was precipitated in a solution containing up to 1 per cent. of nitric acid by means of a 5 per cent. solution of potassium ferrocyanide. Since ferric ferrocyanide is soluble in excess of potassium ferrocyanide, the addition of the latter to the iron solution must be made drop by drop, after a precipitate has once appeared, a reading being taken between each addition of ferrocyanide, and the lowest one being taken, since this represents the maximum opacity.

If y is the percentage of iron in the solution and x the photometric reading, y may be calculated from the empirical formula

$$y = \frac{0{\cdot}03027}{x - 0{\cdot}28}.$$

Using this formula, results were obtained in a series of test analyses correct to 1 part of iron in 1,000,000 of solution, or, with the quantities of iron used, which were generally less than 0·01 gramme, correct to about 1 per cent. on the iron.

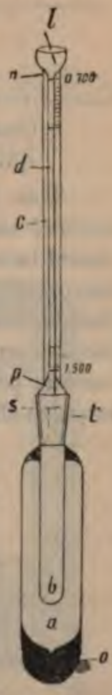
A. G. L.

The Precipitation of Ammonium Vanadate by means of Ammonium Chloride. Arthur Rosenheim. (*Zeits. anorg. Chem.*, xxxii., 181.)—In this reply to Gooch and Gilbert's vindication of Gibbs's method (*supra*), the author states that they appear to have misunderstood his original paper, as he himself obtained sufficiently accurate results when working with pure vanadium solutions, and that his strictures of the method only applied to the case in which tungstic as well as vanadic acid is present, as then not inconsiderable amounts of ammonium tungstate are carried down with the ammonium vanadate. The other investigators mentioned by Gooch and Gilbert also only criticised the method as a means of separating vanadic from molybdic acid.

A. G. L.

APPARATUS.

A New Areo-pyknometer. P. N. Raikow. (*Chem. Zeit.*, 1902, xxvi., 704.)—The apparatus shown in the accompanying diagram possesses several advantages over the areo-pyknometers already devised by Eichhorn and by Vandenvyver, inasmuch as the temperature of the liquid under investigation need not be the same when it is inserted in the bulb as when its density has to be determined. Also it is possible to determine the specific gravity of the liquid on the same portion thereof at different temperatures in succession. The pyknometer is constructed in two parts—a lower, *o t*, and an upper, *p l*. *o* is the usual weight of mercury or shot, *a* is an empty space, and *b* is the liquid reservoir. The upper part consists of a pair of concentric tubes, *d* and *c*, the smaller of which is 1 millimetre in diameter, joined together at their ends, and containing in the annulus the ordinary pyknometer scale. At their base the united tubes become the stopper *p*, through which *d* passes, terminating in a cone to prevent entanglement of air. The stopper is given nearly parallel sides, is about 15 millimetres long, and is very carefully ground in. At the top the inner tube *d* expands into the funnel *l*, which holds about 0·5 c.c. The reservoir *b* is filled with the liquid to the mark *s*, which is in such a position that when the upper part is inserted in the neck the said liquid reaches slightly above the point *n*. The temperature of the whole is then adjusted, and the surplus liquid (that above *n*) is removed with a small pipette or a piece of porous paper. After immersing the apparatus in distilled water and taking the reading, the temperature of the liquid may be raised by placing it in warmer water, removing, as before, the surplus fluid; while if the second reading is to be made at a lower temperature than the first, a drop or two of liquid is added to the funnel to make good the con-



traction. By altering the size of the reservoir, or by omitting it altogether, the apparatus may be made suitable for investigating any desired quantity of liquid.

F. H. L.

The Cause of the Destruction of Platinum Crucibles in Phosphate Analysis. W. C. Heraeus. (*Zeits. f. angew. Chem.*, 1902, xxxvii., 917.)—The destructive agent is phosphorus, which may be liberated under certain circumstances by the reduction of the magnesium phosphate. The author found by experiment that carbon causes the reduction at 950° C., and reducing gases—especially hydrogen—act at a considerably lower temperature. An even more dangerous reducing agent is ammonia. It is very important that this substance be driven off at a moderated temperature before raising the crucible to red heat. Ammonia acts far more energetically on the magnesium phosphate if the latter contain free ammonium phosphate, which is liable to be carried down by the precipitate when ammonia and phosphoric acid are present in excess. This condition of things may often occur at the moment of precipitation, but if the precipitate be allowed to stand some time before filtering, the ammonium phosphate will go into solution again. If the precipitate be dried before being ignited, the drying should be carried out at 100° C., for at 130° or 150° a change takes place whereby phosphoric acid is set free.

A. M.

REVIEW.

CANE-SUGAR AND THE PROCESS OF ITS MANUFACTURE IN JAVA. By H. C. PRINSEN GEERLIGS, Director of the West Java Sugar Experiment Station. Second Edition. (Office of *Cane-Sugar*. Altrincham. Price 5s.)

This is a small, well-written book of about 100 pages, by a most competent author, and, although one would expect from its title and size that its contents would be very limited in their scope, it will be found to supply valuable information useful to chemists engaged in cane-sugar factories in localities other than Java. Besides giving a clear and concise account of the mode of manufacture, there is a large amount of information respecting the composition of the sugar-cane and the various products derived therefrom, and in addition other data of practical importance to the factory chemist. The contents of the book are divided into four parts:

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B. E. R. N.

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Printed for the Society of Public Analysts by Billing & Sons, Ltd., Railway Esplanade, Guildford, England; and published by Baillière, Tindall & Cox, 8, Henrietta Street, Covent Garden, London, W.C., England, to whom all communications regarding Advertisements should be addressed. All other communications to be addressed to the Editor, Westfields, East Grinstead.

MAY 2 - 1921